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The Journal *of the* Society of Dyers and Colourists

Volume 76



Number 11

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FORTHCOMING MEETINGS OF THE SOCIETY

- Thursday, 24th November 1960**
WEST RIDING SECTION. *Fibre Identification—a lecture demonstration.* F. W. Lindley, Esq., Ph.D., and C. Beaumont, Esq., B.Sc., A.R.I.C., A.T.I., A.S.D.C. (Courtaulds Ltd., Droylsden). The Hotel Metropole, King Street, Leeds. 7.30 p.m.
- Thursday, 1st December 1960**
NORTHERN IRELAND SECTION. *The Procion Resin Process.* I. D. Rattee, Esq., B.Sc., A.R.C.S. (Imperial Chemical Industries Ltd.). Grosvenor Rooms, Belfast. 7.30 p.m.
- Friday, 2nd December 1960**
LONDON SECTION. *Dyeing Furnishing Fabrics in London.* R. Hawthorn, Esq. (Henry W. Gibbs Ltd.) (Joint meeting with the London Section of The Textile Institute.) Royal Society, Burlington House, London W.1. 6 p.m.
- Monday, 5th December 1960**
MANCHESTER JUNIOR BRANCH. *High Polymers.* Professor C. E. H. Bawn, C.B.E., F.R.S. (University of Liverpool). Room J/E.11, Jackson Street Extension, College of Science and Technology, Manchester. 4.30 p.m.
- Wednesday, 7th December 1960**
BRADFORD JUNIOR BRANCH. *The Principles of Colour Photography.* C. W. Taylor, Esq. (Ilford Ltd.). Institute of Technology, Bradford. 7.15 p.m.
- Thursday, 8th December 1960**
SCOTTISH JUNIOR BRANCH. *Recent Developments in the Application of Dyes to Polyamide Fibres with special reference to Continuous Processes.* R. E. Fletcher, Esq., B.Sc., A.T.I. (CIBA Clayton Ltd.). Technical College, George Street, Paisley. 7.30 p.m.
- WEST OF ENGLAND AND SOUTH WALES SECTION.** *The Application and Uses of the Synthetic Vinyls in the Textile Industry.* J. H. MacGregor, Esq., Ph.D., F.R.I.C. (Courtaulds Ltd.). Park Hotel, Cardiff. 6.30 p.m.
- Friday, 9th December 1960**
WEST RIDING SECTION. *Half-day Symposium on New Dyeing Techniques.* *The Production of Fast Shades on Polyester Fibre and its Blends.* D. Marian, Esq., M.Sc. (Compagnie Française des Matières Colorantes (Francolor), Paris). *Recent Developments in the Application of Dyes to Polyamide Fibres—"Acid Shock" Process.* R. E. Fletcher, Esq., B.Sc., A.T.I. (CIBA Clayton Ltd.). *The High-temperature Beam Dyeing of Terylene Fabrics.* I. E. Haden, Esq., B.Sc., A.T.I. (Imperial Chemical Industries Ltd.). Institute of Technology, Bradford.
- Tuesday, 13th December 1960**
SCOTTISH SECTION. *Modern Flow Principles and Machine Design.* (With particular reference to package and high-temperature beam dyeing.) G. S. Helliwell, Esq. (Samuel Pegg & Son Ltd.). St. Enoch Hotel, Glasgow. 7.15 p.m.
- Thursday, 15th December 1960**
MANCHESTER SECTION. *Ladies Evening. Carpets.* The Great Hall, College of Science and Technology, Manchester. 7 p.m.
- MIDLANDS SECTION.** *Half-day Symposium on Chemical Finishing.* *Stimulus and Response.* G. W. Madaras, Esq., Ph.D. and S. N. Bradshaw, Esq. *Easy Care Finishes on Viscose Rayon.* D. H. Ogden, Esq. B.Sc.(Tech.), A.M.C.T., A.R.I.C. (British Rayon Res. Assoc.). *Lubricants in Resin Finishing.* R. W. Williamson, Esq. F.T.I., F.S.D.C. (Hickson & Welch Ltd.). *Chemistry of Resins.* A. R. Smith, Esq., B.A., B.Sc., F.R.I.C. (B.I.P. Chemicals Ltd.). Charge for symposium 2s. 6d. including afternoon tea. Daybrook House, Daybrook, Nottingham. 2.15 p.m.
- Tuesday, 20th December 1960**
HUDDERSFIELD SECTION. *The Dyeing of Courtelle.* J. S. Ward, Esq., B.Sc. (Courtaulds Ltd.). Silvios Cafe, Huddersfield. 7.30 p.m.
- Tuesday, 10th January 1961**
NORTHERN IRELAND SECTION. *Standards for Consumer Requirements.* J. S. Ingham, Esq., M.Sc., F.R.I.C., F.S.D.C. (Marks and Spencer Ltd.). Grosvenor Rooms, Belfast. 7.30 p.m.
- Thursday, 12th January 1961**
SCOTTISH SECTION. *Recent Developments in Wool Dyeing.* D. G. Evans, Esq., B.Sc. (CIBA Clayton Ltd.). Scottish Woollen Technical College, Galashiels. 7.30 p.m.
- WEST RIDING SECTION.** *Modern Piece Scouring Methods in Relation to Uneven Dyeing.* B. F. J. Moxon, Esq. (Wool Industries Research Association). The Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.
- Friday, 13th January 1961**
LONDON SECTION. *Fibre Identification—a lecture demonstration.* F. W. Lindley, Esq., Ph.D., and C. Beaumont, Esq., B.Sc., A.R.I.C., A.T.I., A.S.D.C. (Courtaulds Ltd., Droylsden). (Joint meeting with the London Centre of the Guild of Dyers and Cleaners.) Royal Society, Burlington House, London W.1. 6.30 p.m.
- Monday, 16th January 1961**
BRADFORD JUNIOR BRANCH. *Moths and Mothproofing.* A. Gabriel, Esq., B.Sc. (Shell Chemical Co. Ltd.). Institute of Technology, Bradford. 7.15 p.m.
- Tuesday, 17th January 1961**
HUDDERSFIELD SECTION. *Defending the Frontiers of Civilisation.* J. David, Esq., B.Sc.(Tech.), A.M.C.T., A.T.I. (Catomance Ltd.). (Joint meeting with Halifax Textile Society.) Silvios Cafe, Huddersfield. 7.30 p.m.
- LEEDS JUNIOR BRANCH.** *Moths and Mothproofing.* A. Gabriel, Esq., B.Sc. (Shell Chemical Co. Ltd.). The Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, Leeds 2. 3.15 p.m.
- SCOTTISH SECTION.** *Continuous Dyeing of Synthetic Fibres and Their Blends with Other Fibres.* J. Khacheyan, Esq. (C.F.M.C.). St. Enoch Hotel, Glasgow. 7.15 p.m.
- Wednesday, 18th January 1961**
MIDLANDS SECTION. (Title later). G. H. Lister, Esq., Ph.D. (Sandoz Ltd.). King's Head, Loughborough. 7 p.m.
- Thursday, 19th January 1961**
MANCHESTER SECTION. *Printing of Synthetic Fibres.* R. E. Fletcher, Esq., B.Sc., A.T.I. (CIBA Clayton Ltd.). Lecture Theatre, Manchester Literary and Philosophical Society, 36 George Street, Manchester 1. 7 p.m.
- MANCHESTER JUNIOR BRANCH.** *Fibre Identification.* F. W. Lindley, Esq., Ph.D. and C. Beaumont, Esq., B.Sc., A.R.I.C., A.T.I., A.S.D.C. (Research Dept., Courtaulds Ltd.). Room J/E.11, Jackson Street Extension, College of Science and Technology, Manchester. 4.30 p.m.
- Friday, 20th January 1961**
WEST RIDING SECTION. *Annual Dinner and Dance.* Victoria Hotel, Bradford.
- Saturday, 21st January 1961**
MANCHESTER SECTION. *Annual Dinner and Dance.* Jubilee Suite, Grand Hotel, Manchester. 6 p.m. for 6.30 p.m.
- Thursday, 26th January 1961**
WEST RIDING SECTION. *New Light on the Reactivity of Wool with Acids and Alkaline Solutions.* R. L. Elliott, Esq., B.Sc., Ph.D. (London), F.R.I.C., F.T.I., F.S.D.C. and R. S. Asquith, Esq., M.A., Ph.D., F.S.D.C. (Bradford Institute of Technology). The Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.
- Friday, 27th January 1961**
LONDON SECTION. *Annual Dinner and Dance.* Waldorf Hotel, Aldwych, London W.C.2. 7 p.m. for 7.30 p.m.
- Monday, 30th January 1961**
HUDDERSFIELD SECTION. *Modern Piece Scouring Methods in Relation to Uneven Dyeing.* B. F. J. Moxon, Esq. (Wool Industries Research Association). (Joint meeting with the Huddersfield Textile Society.) College of Technology, Huddersfield. 7.30 p.m.

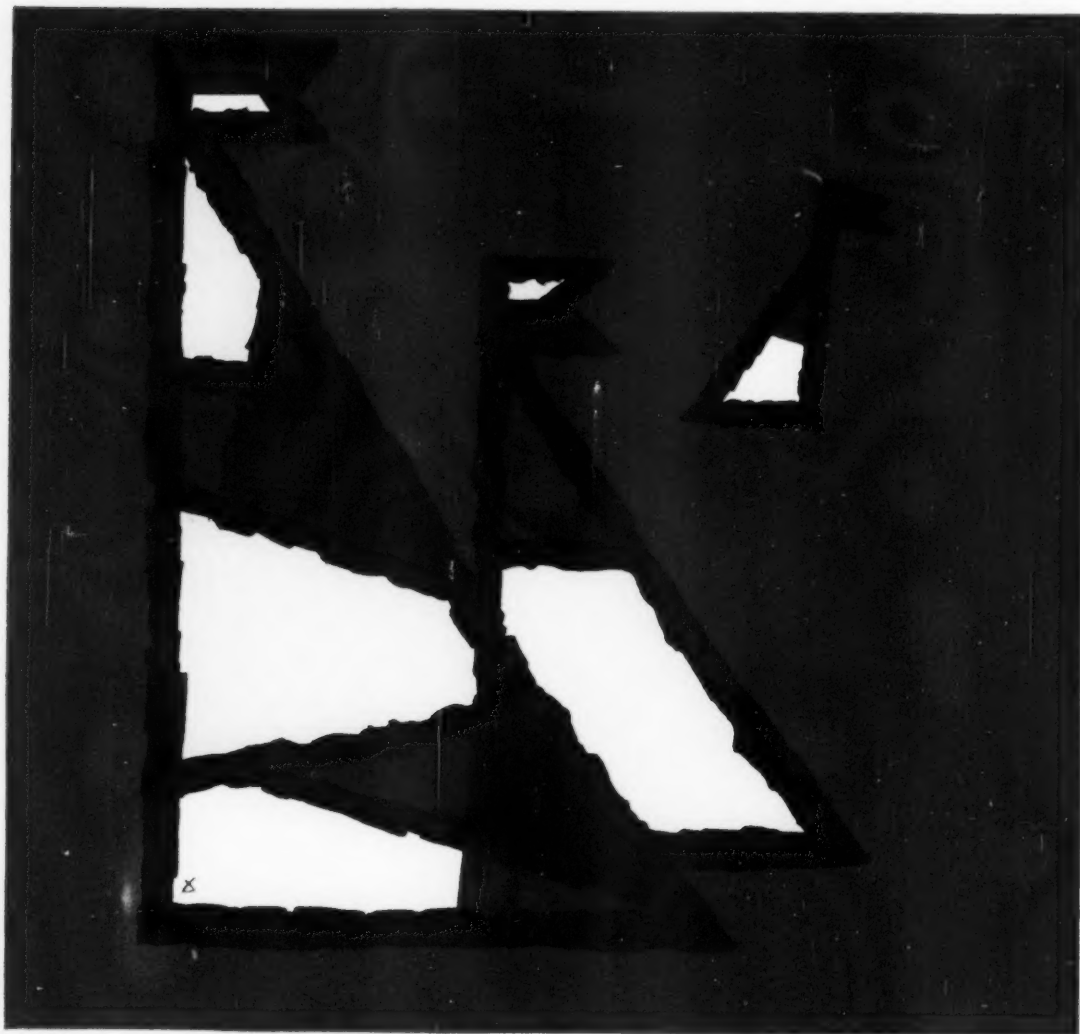


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FORTHCOMING MEETINGS OF THE SOCIETY — continued from page iv

Friday, 3rd February 1961

LONDON SECTION. *Dyeing of Millinery and Allied Materials.* Short papers by members of Barford Bros. Ltd., Luton. Royal Society, Burlington House, London W.1. 6 p.m.

Tuesday, 7th February 1961

NORTHERN IRELAND SECTION. *Science in the Detection of Crime.* Dr. A. J. Howard (Director of the Department of Industrial Forensic Science). Joint meeting with the Textile Institute. Chamber of Commerce Board Room, Donegall Square West, Belfast. 7.30 p.m.

Wednesday, 8th February 1961

MIDLANDS SECTION. *Work Study in the Dyehouse.* B. Lockwood, Esq., B.Sc., and R. W. Richardson, Esq., Ph.D., B.Sc., A.R.I.C. (Furzebrook Knitting Co. Ltd.). College of Art and Technology, Leicester. 7 p.m.

Thursday, 9th February 1961

WEST OF ENGLAND AND SOUTH WALES SECTION. *Trade Effluents—The Legal Position and Method of Treatment.* J. H. Harwood, Esq., B.Sc., A.R.I.C. (Peter Spence & Sons Ltd.). County Hotel, Taunton. 6.30 p.m.

WEST RIDING SECTION. *The Problem of Illumination in Colour Matching.* K. McLaren, Esq., B.Sc., F.R.I.C., F.S.D.C. (Imperial Chemical Industries Ltd.). The Hotel Metropole, King Street, Leeds. 7.30 p.m.

Tuesday, 14th February 1961

BRADFORD JUNIOR BRANCH. *Modern Piece Scouring Methods in Relation to Uneven Dyeing.* B. F. J. Moxon, Esq. (Wool Industries Research Association). Institute of Technology, Bradford. 7.15 p.m.

NORTHERN IRELAND SECTION. *The Principles of Colour Photography.* D. P. Ayres, Esq., B.Sc., A.R.P.S. (Ilford Ltd.). Grosvenor Rooms, Belfast. 7.30 p.m.

SCOTTISH SECTION. *Mechanism of Crease-recovery.* J. A. Wilson, Esq., B.Sc., Ph.D. (British Rayon Research Assoc.). (Joint meeting with the Textile Institute.) Room M 406, Royal College of Science and Technology (New Building) Glasgow. 7.30 p.m.

Thursday, 16th February 1961

MANCHESTER JUNIOR BRANCH. *Three short papers by members of the Section.* (Prize donated by Manchester Senior Section.) Room J/E.11, Jackson Street Extension, College of Science and Technology, Manchester. 4.30 p.m.

Friday, 17th February 1961

MANCHESTER SECTION. *The Dye and the Fibre—Study of the Movement of Dye Molecules Within the Fibre.* Dr. R. McGregor (Lecturer in Textile Chemistry, The University of Manchester). Lecture Theatre, Manchester Literary and Philosophical Society, 36 George Street, Manchester 1. 7 p.m.

Tuesday, 21st February 1961

HUDDERSFIELD SECTION. *Some Aspects of the Chemistry of Afterchroming.* L. Peters, Esq., Ph.D., M.Sc. (Leeds University). Silvios Cafe, Huddersfield. 7.30 p.m.

Thursday, 23rd February 1961

WEST RIDING SECTION. *Recent Developments in Dyeing Acrylic Fibres.* B. Kramrisch, Esq., F.R.I.C., F.T.I., F.S.D.C. (CIBA Clayton Ltd.). The Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

Friday, 24th February 1961

LONDON SECTION. *Technical Aspects of Textile Marketing in Relation to the Dyeing and Finishing Industry.* H. A. Thomas, Esq., Ph.D., F.R.I.C., F.T.I., F.S.D.C. (Marketing Division Director, Courtaulds Ltd.). George Hotel, Luton. 6.30 p.m.

Monday, 27th February 1961

HUDDERSFIELD SECTION. *Application of Colour Theory to Fibres, Yarns and Fabrics.* P. Warburton, Esq., M.C., F.S.A.M. (Former Head of Bury Municipal School of Arts and Crafts). Joint Meeting with the Halifax Textile Society. Alexandra Hall, Halifax. 7.30 p.m.

Tuesday, 28th February 1961

LEEDS JUNIOR BRANCH. *The Applications and Uses of the Synthetic Vinyls in the Textile Industry.* J. H. MacGregor, Esq., Ph.D., F.R.I.C. (Courtaulds Ltd., Bocking). The Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, Leeds 2. 3.15 p.m.

Friday, 3rd March 1961

LONDON SECTION. *Colour Measurement: its possibilities for the Colourist.* H. W. Ellis, Esq., Ph.D., A.R.C.S., A.R.I.C., F.S.D.C. (CIBA Clayton Ltd.). Royal Society, Burlington House, London W.1. 6 p.m.

MIDLANDS SECTION. Annual Dinner. Nottingham Co-operative Society, Upper Parliament Street, Nottingham. 7 p.m.

Tuesday, 7th March 1961

LEEDS JUNIOR BRANCH. *The Principles of Colour Photography.* D. P. Ayres, Esq., B.Sc., A.R.P.S. (Ilford Ltd.). The Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, Leeds 2. 3.15 p.m.

NORTHERN IRELAND SECTION. *The Finishing of 'Terylene' Flax Fabrics.* I. E. Haden, Esq., B.Sc., A.T.I. (Imperial Chemical Industries Ltd.). Grosvenor Rooms, Belfast. 7.30 p.m.

Thursday, 9th March 1961

MANCHESTER JUNIOR BRANCH. Annual General Meeting. *The Standfast Molten Metal Dyeing Machine.* Lecture and Film. A. Ogden, Esq. (Standfast Dyers and Printers Ltd.). Room J/E.11, Jackson Street Extension, College of Science and Technology, Manchester. 4.30 p.m.

MIDLANDS SECTION. *Flameproofing.* J. E. W. Perfect, Esq., B.Sc. (Proban Ltd.). (Joint meeting with the Coventry Textile Society). Courtaulds Acetate and Synthetic Fibres Laboratory, Lockhurst Lane, Coventry. 7 p.m.

WEST RIDING SECTION. *Stimulus and Response—Reactions to Some Current Problems in Textile Finishing.* G. W. Madaras, Esq., Ph.D., and S. N. Bradshaw, Esq. (Courtaulds Ltd., Coventry). The Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

Tuesday, 14th March 1961

NORTHERN IRELAND SECTION. *General Principles in the Screen Printing of Fabrics made from Natural and Synthetic Fibres.* P. Pyle, Esq., A.R.I.C. (Decorative Fabrics Ltd.). Grosvenor Rooms, Belfast. 7.30 p.m.

SCOTTISH SECTION. (Title later). G. H. Lister, Esq., Ph.D. (Sandoz Ltd.). St. Enoch Hotel, Glasgow. 7.15 p.m.

Wednesday, 15th March 1961

BRADFORD JUNIOR BRANCH. *The Dyeing of Worsted Pieces.* H. S. Smith, Esq. (CIBA Clayton Ltd.). Institute of Technology, Bradford. 7.15 p.m.

MIDLANDS SECTION. *Early Attempts to Dye Secondary Acetate.* E. Stanley, Esq., M.Sc., and A. J. Wesson, Esq. (British Celanese Ltd.). Celanese Sports Pavilion, Spondon. 7 p.m.

Friday, 17th March 1961

MANCHESTER SECTION. *Textile Finishing Symposium.* (Details to be announced later). Great Hall, College of Science and Technology, Manchester. 2 p.m.

Tuesday, 21st March 1961

HUDDERSFIELD SECTION. *Fibre Identification—a lecture demonstration.* F. W. Lindley, Esq., Ph.D. and C. Beaumont, Esq., B.Sc., A.R.I.C., A.T.I., A.S.D.C. (Courtaulds Ltd.). Silvios Cafe, Huddersfield. 7.30 p.m.

Thursday, 23rd March 1961

MIDLANDS SECTION. *The Dyeing of Blends for Use in Carpets.* R. C. Cheetham, Esq., A.M.C.T., F.R.I.C., F.S.D.C., (Courtaulds Ltd.). Carpet Trades Ltd., Canteen, Kidderminster. 7 p.m.

WEST OF ENGLAND AND SOUTH WALES SECTION. *Technical Service in the Dyestuffs Industry.* F. North, Esq., B.Sc. (Imperial Chemical Industries Ltd.). The Pillar Room, Mauretania, Bristol. 6.30 p.m.

WEST RIDING SECTION. Annual General Meeting. Followed by *Four Decades with Dicot.* A. J. Wesson, Esq. (Furzebrook Knitting Co. Ltd.). The Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

continued on page viii

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Readers requiring general information regarding the Official Notices, List of Officers of the Society, etc, should consult pages 1-10 of the January 1960 and pages 389-396 of the July 1960 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (Telephone Bradford 25138). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*

LECTURE

The Dyeing and Finishing of Fabrics containing Tricel Triacetate Fibre

R. J. Mann

COMMUNICATION

The Mechanism of Dyeing with Procion Dyes

I—The Mechanism of Alkali Adsorption by Cellulose

H. H. Sumner

FORTHCOMING MEETINGS OF THE SOCIETY—continued from page vii

Friday, 24th March 1961

LONDON SECTION. Annual General Meeting and Dinner. Waldorf Hotel, Aldwych, London W.C.2. 6.30 p.m. for 7 p.m.

Tuesday, 28th March 1961

SCOTTISH SECTION. Ladies Evening. *Fashion and Mistress Brown, 1780-1880*. R. A. Peel, Esq., F.S.D.C. (Subject will be women's clothes, the colours and dyes, the influence of events—French Revolution, Chartist Movement, Queen Victoria, Mauve and Magenta (i.e. "Coal-Tar" dyes), Church of Scotland, Episcopal Church, etc.—upon the dress of Scotswomen). St. Enoch Hotel, Glasgow. 7.15 p.m.

Tuesday, 11th April 1961

SCOTTISH SECTION. Annual General Meeting. 7 p.m. Followed by *An Instrumental Approach to Colour Matching*. R. Sinclair, Esq., B.Sc., A.S.D.C. at 7.30 p.m. St. Enoch Hotel, Glasgow.

Thursday, 13th April 1961

MIDLANDS SECTION. Annual General Meeting. *Calculations of Fading Rates of Dyes of Different Lightfastness on Exposure to Fluorescent Tubes*. S. M. Jaekel, Esq., B.Sc., A.R.C.S., A.R.I.C. and C. D. Ward, Esq., B.Sc. *Effect of Dry Heat Treatment on the Dye Uptake of Nylon*. S. M. Jaekel, Esq., and D. A. Bampton, Esq., B.Sc. King's Head, Loughborough. 7 p.m.

Tuesday, 18th April 1961

HUDDERSFIELD SECTION. Annual General Meeting. *The Identification of Dyes in Substance and on the Fibre*. G. W. Midgelow, Esq., B.Sc. (Imperial Chemical Industries Ltd.). (Joint meeting with the Huddersfield Section of the Royal Institute of Chemistry.) Silvios Cafe, Huddersfield. 7.30 p.m.

Thursday, 20th April 1961

MANCHESTER SECTION. Annual General Meeting. *Factors Affecting the Efficiency of the Washing Process*. G. J. Parish, Esq., B.Sc., A.Inst.P. (British Cotton Industry Research Association). Lecture Theatre, Manchester Literary and Philosophical Society, 36 George Street, Manchester 1. 7 p.m.

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THE JOURNAL

OF THE

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Proceedings of the Society

THE SIXTH GEORGE DOUGLAS LECTURE

Wool as a Fibre in Dyeing and Finishing

A. B. D. CASSIE

*Meeting held at the Midland Hotel, Bradford, on 13th October 1960,
Mr. F. M. Stevenson (President of the Society) in the chair*

The importance of the setting of wool fibres on packages is discussed in relation to cloth manufacture and shrinkage in finishing. Some remarkable changes that come about in low-temperature dyeing of wool when fibres are cleaned with various solvents are described.

INTRODUCTION

This is the sixth George Douglas Memorial Lecture and I was greatly honoured when Mr. Morton Wood invited me to deliver it. He suggested that I might choose as a subject "Wool as a Fibre in Dyeing and Finishing" and I am very happy to have built the lecture around this theme.

This is the first Lecture to deal with a single fibre, and you will notice, too, that the choice of the lecturer is somewhat peculiar, in that I am a physicist by training and the choice of a physicist may seem a little strange when one reflects how dyeing and finishing is generally taken to be the preserve of the chemist. I feel this choice must have come about because Mr. Wood and his committee realised that George Douglas had much to do with physics in his life's work.

I much regret that I never met George Douglas during his lifetime, and I have had to rely on information from friends and from the literature to learn something of his remarkable character and ability.

He began his working life with A. & S. Henry, right at the centre of textiles. However, at 19 he entered the Yorkshire College, now Leeds University, and from there he went to Edward Ripley & Sons, Bowling Dyeworks. He soon reorganised this firm, using, I am sure, many physical principles in doing so. In 1898 he was instrumental in forming the Bradford Dyers' Association to help rationalise the piece dyeing and finishing industry. It became, under his guidance, the largest piece dyeing and finishing company in the world. He was Chairman from 1924-45, and was Chairman and sole Managing Director from 1938-45. He chose as his home Farfield Hall, and

anyone who has admired the delightful setting of this house will realise that George Douglas had a keen appreciation of all beautiful things.

Interest in technical developments continued throughout his long life, and he served the Society well, being Vice-President from 1894-1912, and President from 1912-14. He was made an Honorary Member in 1934. Thus, George Douglas served the industry through the period when man-made fibres entered commerce, and although he must have spent much time devising methods of dyeing and finishing these fibres and cotton, there is little doubt that wool must always have been one of his main preoccupations. Dr. Levinstein has recorded, in the first Memorial Lecture, the lively changes that took place in dyes and dyeing in George Douglas's lifetime, and at Ripley's and later at B.D.A. George Douglas kept Bradford to the fore in using these new discoveries. The discoveries were chemical, but to use them efficiently must have called for much physical insight. We must pay tribute to those who looked after the physical problems so well when, I am sure, the underlying physical principles were so little understood. After all, heat exchange, dye exchange, the movement of liquors through cloth, the action of nips, and drying, are all physical phenomena. I often think that a physicist would be the man to tackle these problems—though, of course, we would then call him a chemical engineer!

WOOL AS A FIBRE IN FINISHING

Setting

Here I will not discuss topics of general application to all fibres; rather I will deal with the physics of wool and how that enters into finishing.

Wool has many outstanding properties for finishing: the power of absorbing molecules (as in dyeing), felting, and setting are those that come most readily to mind. I would like to deal first with the unique setting properties, because we now take them for granted, yet if we search the literature we find that the first systematic study was published by Astbury and Woods in 1933. Yet

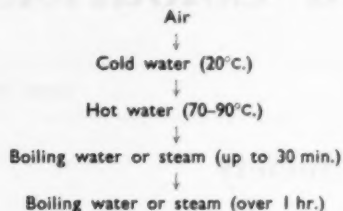


FIG. 1—Setting of wool fibres

who can doubt that George Douglas and those who worked with him at Ripley's and at the B.D.A. used, to a very great extent, the setting properties of wool to enhance the quality and beauty of their finished fabrics? But that is not all, for although Astbury and Woods revealed for the first time how wool could be set or supercontracted in steam, it was not until the paper by Blackburn and Lindley was published in the Society's *Journal* in 1948 that the effect of time on setting was realised. Yet this effect is most important for fabric construction and finishing, and here again those at Ripley's and B.D.A. must have made effective use of the properties for many years before their fundamental characteristics were revealed.

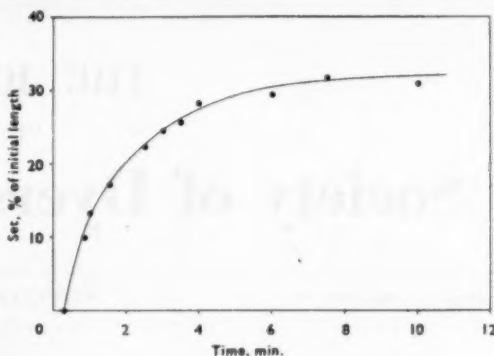


FIG. 2—Cold-water set v. time of steaming

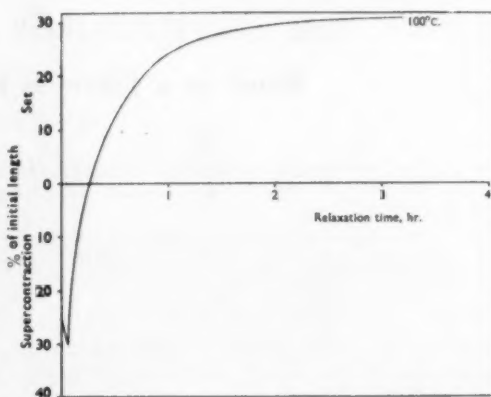


FIG. 3—Steam set v. time of steaming

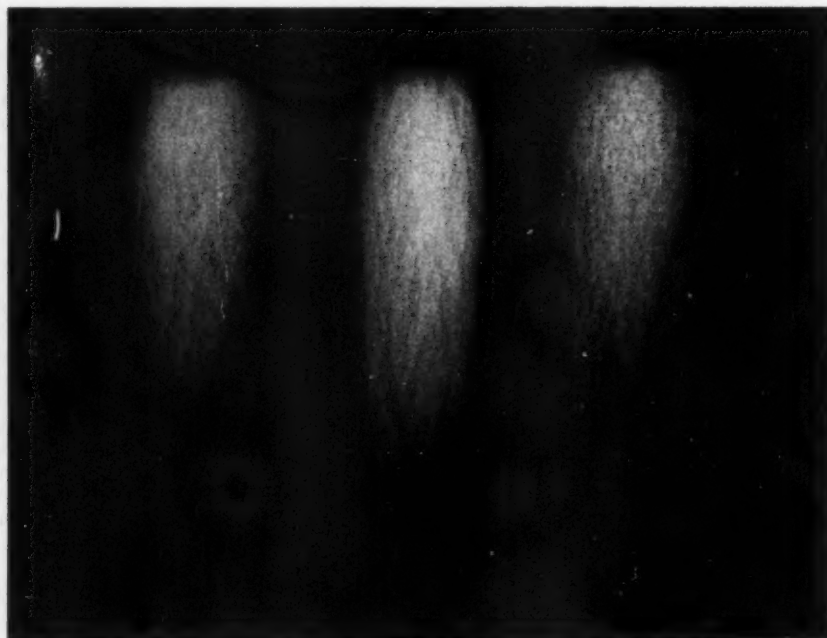


FIG. 4

Setting of wool is a most interesting and complex phenomenon; Fig. 1 indicates how any set may be released by subsequent processing. It appears that, given time, wool can be set to any desired length, at moderate temperatures, provided it is wet or has a sufficiently high moisture content. Thus, if we set wool in water at 20°C., the set can be released by immersing the wool in water at a higher temperature, and so on up the scale, until, of course, we reach the permanent set given by extended steaming or chemical treatment. Such set, as every woman knows from the permanent setting of hair, is very difficult—if not impossible—to remove. But apart from the effect of temperature, there is also the time factor, which is a most important one for fabric finishing. This effect has been studied only for the severe case of setting in steam, and Fig. 2 and 3 show the effect of time of setting by steam on the release of set. If we choose cold water as the medium for releasing set, steaming for about six minutes gives what might be termed "a set permanent to cold-water release". On the other hand, steaming for over an hour is required to give a set which is permanent to release in steam.

These results relate to setting in steam, but similar time effects arise when fibres are set at high humidities or in cold water, although no systematic study has been made. These are the sets that are so important for fabric finishing, and I am thinking here not of setting a fibre that has been extended in length by 30 or 40%, but rather of a fibre from which merely the crimp or part of the crimp has been removed. The preparation of a top, for example, imposes small strains on all the fibres and tends to remove the crimp. The fibres, when in the form of a top ball, are unable to recover their crimp because a ball is a solid structure; after being stored for some time the fibres acquire a set which can readily be released in water, or more strikingly by chemicals such as sodium bisulphite. Fig. 4 shows this for a top that we were using in studying shrink-resist treatments. The middle draw is that for the top before experimental work began; that on the right is the same top after immersion in bisulphite solution, and that on the left after shrink-resist treatment. Clearly, a spinner judging the length of these tops would regard the centre one as much superior, but, in fact, fibre length measurements of the three specimens showed that the actual lengths of fibres in them are almost identical. The difference in length of the draw is entirely due to the recovery of crimp by release of the set of the original top in the chemical solution. This may seem a severe treatment for a top, but it is becoming of importance now that attempts are being made to shrink-resist wool at the top or backwashing stage of processing. Such a change in the fibre diagram would, no doubt, be of importance in spinning.

Fig. 5 shows a similar phenomenon at a later stage of processing, namely after spinning. Two hanks were reeled from the same lot of yarn which had been stored on spinning bobbins for some time. A spinning bobbin is certainly an ideal

medium for maintaining fibres in a stretched condition, because it is an even harder package than a top, and so the fibres have less chance to recover their crimp and so change their length. The two hanks were wound on the same reel and one of them was placed in a cool damp cellar for a few days whilst the other was left in the office atmosphere. The hank that was cellared quickly recovered and is seen in Fig. 5 to be considerably shorter than

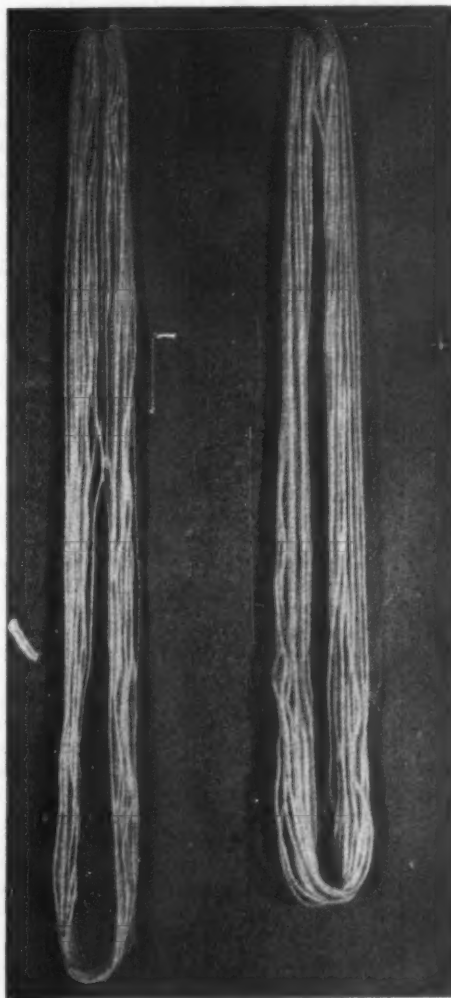


FIG. 5

the other, which retained its reeled length. This illustrates again the setting power of wool and how set is retained in a dry atmosphere but may be released in a damp one, if the fibres and yarns are in a sufficiently free state for the reduction in length to take place.

Normally the yarns would be woven into fabric in a dry atmosphere and so would have the length shown by the longer hank, but after the fabric had been wetted out in a process such as scouring,

the yarns would attempt to return to the shorter length of the released hank. This again shows the great importance of setting that takes place with time—setting, I think, of the fibres in a slightly uncrimped condition. But on being wetted out, the fibres make a considerable effort to return to their fully crimped form; this tendency, added up over the large number of fibres in a fabric, means that considerable shrinkage forces can arise. In fact, 0.8 g. tension is required to remove the crimp from a single merino fibre; this force summed over, say, 100 fibres (as would be found in a 2/48s yarn) can give a retractive force of 80 g., which is much more than the tension at which such yarns would be warped. Over a warp of 3,000 ends, the load would be 600 lb. Crimping forces, their elimination by setting, and their reappearance on wetting can therefore exert considerable loads within a cloth structure when aggregated over the many fibres in a fabric.

Effect of Set on Fabric Dimensions

If there is a dependence of set on time, there should be a difference between fabrics made from "red-hot" tops and rovings and those made from yarns where the tops or rovings have been stored for long periods.

We have recently carried out experiments at Torridon to see if this difference does exist. Fig. 6 shows some typical results, and it is noteworthy that the fabrics made from stored tops and rovings retract to a shorter length; measurements of width show the same effect. This is what we might expect, for tops and rovings are stored as solid packages and the fibres have no opportunity to retract in length during storage. Temporary set appears if fibres are held extended for a long enough period, even at ordinary temperatures and humidities. The fibres obviously retain this extension all through the warping and weaving processes, and retraction occurs only on scouring.

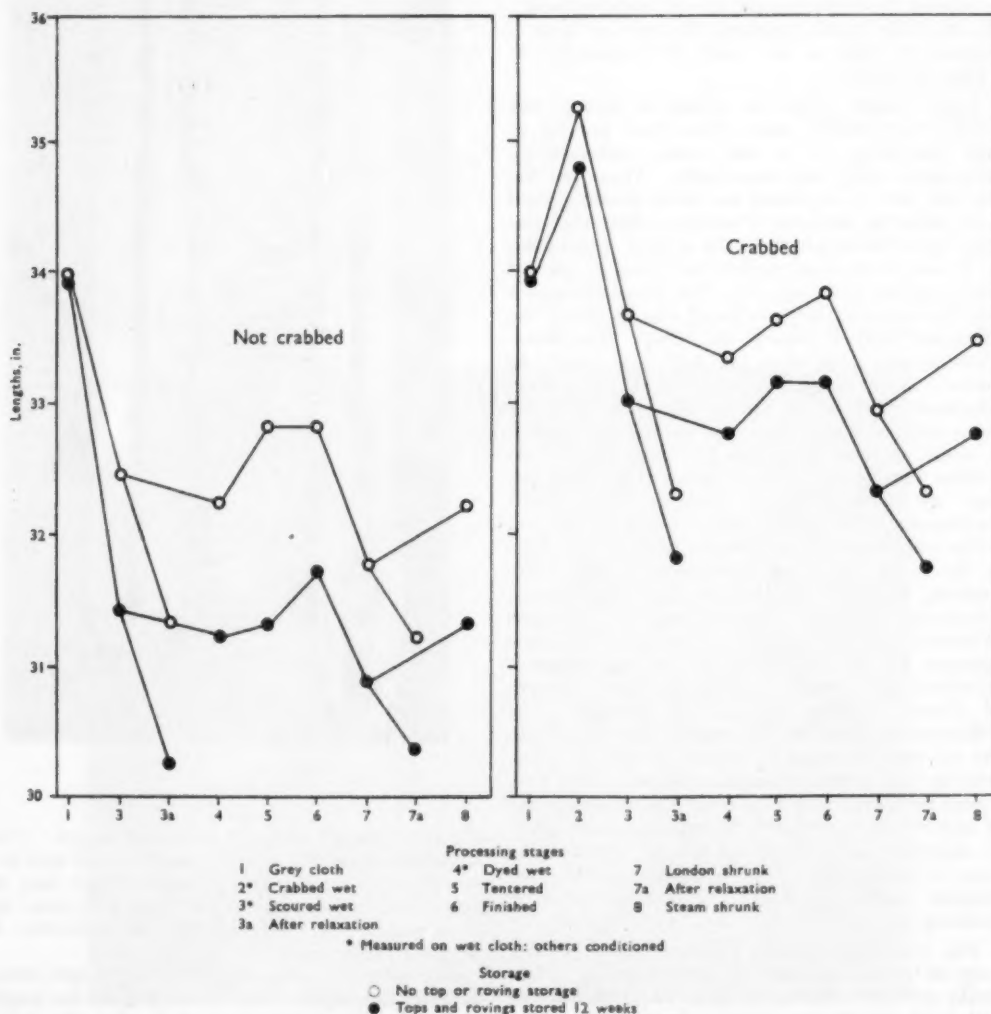


FIG. 6—Effect of storage and crabbing on fabric length

Results for both crabbed and uncrabbed pieces are included in Fig. 6. Crabbing does set the fabric at a greater length, but the difference between stored and unstored materials seems to persist.

These results, when first presented to our technical committee, were thought to be wrong, but reflection about what happens to wool during storage, as I have indicated, suggested dimensional changes in the direction observed. However, as the feeling in the industry was contrary to these results, we repeated the experiments; the results were similar to those that I have reported. More recently we have measured the retraction of yarns on wetting out after different periods of top, roving, and yarn storage, since these phenomena should be visible in the yarns before they are woven. This is in fact so, but recent results seem to indicate that if the fibres are stored for really long periods—say six months to a year—it is then difficult to recover the strains even by wetting in water.

Fiddle-strings

One conclusion of these experiments is that excessive shrinkage of cloth cannot be attributed to lack of storage of the fibres; in fact, storage tends to give greater shrinkage in finishing and will lead to a cloth that feels better built. It is not difficult to imagine that, if differences of this extent arise merely by storing the fibres at reasonable humidities, very big differences may arise if the yarns are stored wet. This can happen when conditioning of yarns by liquid water is resorted to, for if anything goes wrong with the conditioning unit, or if a yarn is left in a trough for too long—particularly if a wetting agent is present—the section of wet yarn becomes unduly stretched in winding and a temporary set is obtained when the yarn is held on a hard package for any period. When this section of yarn is woven into a fabric and is subsequently wetted out, it tends to retract to its original length, giving rise to the now familiar "fiddle-string".

This particular defect was very troublesome when wool-Terylene yarns were first introduced, and it is interesting to discover how this arose. It arose because the great strength of Terylene made possible very large extensions of the yarn without breakage, and these sections of yarn which had been so much extended were wound on to hard packages where the wool acquired a temporary set. The extended length was maintained throughout the weaving process, but as soon as the fabric was wetted out the wool retracted to its original length, giving a very striking "fiddle-string", or "shiner" as it is called in America. By dyeing the wool red and leaving the Terylene undyed, it was conclusively shown that, although the Terylene provides the strength required for excessive stretch of the yarns, it is, in fact, the wool that causes the retraction.

Setting for Finish

These are some of the interesting phenomena that result from the temporary setting properties of wool, and with which dyers and finishers must have coped long before the detailed behaviour of

the fibres was known. Some of them, I am afraid, lead to blemishes, but on the other hand the setting properties of wool—particularly the permanent setting properties—are used to very great effect in finishing. I have already mentioned crabbing, which can maintain the dimensions of a piece, although it is sometimes held to spoil the handle of a top-quality worsted fabric. Setting by blowing is another example where the finisher makes use of this interesting setting property of wool, and finally there is the beautiful finish put on by the paper press.

This is an interesting effect because the cloth is put into the press at a fairly high moisture content and, because of the nature of the press, water vapour cannot evaporate, even though the cloth is heated considerably. The high temperature and high moisture content tend to give a permanent finish to the surface of the fabric. Much of the great appeal of West Riding cloths comes from this pressing process. It is a finish that is very nearly permanent, but steaming, as practised by some spongers in America, does tend to spoil it, no doubt by raising the temperature and moisture content of the cloth above that which applied in the press and so releasing the set. But if drastic after-finishing treatments are avoided the set given by a paper press can be remarkably durable.

Bulk despite Setting

Setting, then, is one of the remarkable physical properties of wool, and you may feel that having made use of it—particularly in pressing—a smooth, solid fabric is the product of the finisher's work. In fact, this is not so, because wool has another remarkable physical property, namely its bulking tendency. Wool fibres are exceedingly difficult to pack closely; in fact, the densest packing that normally arises is in worsted yarn, where some attempt has been made to parallelise the fibres and persuade them to pack as closely as possible, but even here 60% of the volume of the yarn is air, and only 40% is the wool fibre substance. Apparently, no matter how hard you try to pack the fibres more closely, by pressing or even by felting—which is another remarkable physical phenomenon—the ratio of air to fibre cannot readily be decreased. It is this bulking property that enables us to finish wool fabrics so that they appear solid, yet remain open in structure and comfortable just because of their air content. The bulking property of the fibres results, I feel sure, from their peculiar elastic constants. Compared with other fibres, it is much easier to stretch than to twist a wool fibre, and it is this resistance to torsion that, together with crimp, produces bulking properties.

DYEING

The unique setting property of wool is all-important for finishing, but, of course, a great deal of the beauty of wool fabrics comes from their colour, and we must therefore look at how the fibre behaves in absorbing dyes and so producing many beautiful designs of fabric. Wool is well known for its moisture absorption. To some this

TABLE I
Rates of Uptake of Naphthalene Orange G at 30°C. by Merino Wool Soxhlet-extracted with Petroleum Ether

Experiment	Additional pretreatment	Dyeing solvent	$\alpha \times 10^{-2}$ cm. min. ⁻¹	$D \times 10^{-12}$ cm. ² min. ⁻¹
1	None	Water	1.6	4.8
2	Ethanol (Soxhlet)	Water	10.7	120
3	Butanol solution, ether	Water	14	153
4	Lissapol N solution	Water	2.7	50
5	Butanol solution, ether	Butanol solution	35	3,400
6	Prolonged butanol solution, no ether	Butanol solution	8.7	—

is a nuisance, as in commercial transactions where corrections for regain have to be applied. In other fields it is a most valuable property. It is, for example, certainly responsible for much of the comfort given by wool clothing, and it is, too, important in dyeing. I do not think we realised the value of the water-absorbing powers of wool for dyeing until the advent of man-made fibres which absorbed very little water and proved correspondingly difficult to dye.

Wool has, I believe, always been one of the easiest fibres to dye. This is no doubt largely because of its chemical composition, but there is, too, the fact that the inside of the wool fibre is easily accessible, which means that dye molecules can enter the fibre and be fixed there to give colours that are fast to many agencies encountered during wear. It is, however, only in recent years that the absorptive powers of wool have been studied from the point of view of molecules other than water, and some interesting phenomena have appeared as a result.

One of the most striking is, I think, the effect of organic solvents as aids to dyeing. One early theory of how solvents speeded up dyeing, particularly at low temperatures, was that the dye was preferentially dissolved in the solvent, which in turn was preferentially attracted to the wool fibres, forming a solvent dyebath of high concentration around each fibre. Inspection of such a theory reveals, however, that it is not likely to be the complete story. If the dye molecules are preferentially attracted to a solvent layer round the wool, they will be reluctant to pass from this attractive solvent into the wool fibre. Arguments based on thermodynamics show that this is so to such an extent that the concept of a concentrated dyebath around each fibre does not lead to increased rates of dyeing. The cause of the effect is likely to be revealed amongst the complex effects that arise when molecules diffuse into a material such as wool. There is ample evidence now that two factors control this diffusion. The first is a barrier, possibly in the form of an epicuticle, which encases the fibre proper and through which it is difficult for these larger molecules to pass; secondly, when the molecules have passed through the barrier they have to diffuse through the material of the fibre into its very centre if dyeing is to be satisfactory. A study of these two factors—the surface resistance of the fibre and the diffusion within the fibre itself—is now revealing several interesting features of wool.

Table I shows some of these results in terms of a surface admittance factor (α) which takes into account the outer barrier of the fibre and the diffusion coefficient within the fibre itself. The scientist always starts with clean wool, by which he means root ends of wool fibres; this avoids any troubles that may arise because of the weathering of fibre tips. Then to make sure that the fibres are quite clean he normally extracts them with petroleum ether to remove grease and follows this by thorough rinsing in distilled water to remove any trace of petroleum ether. The results of Experiment 1 were obtained with these "clean" fibres. In Experiment 2 the fibres were Soxhlet-extracted for several hours with ethanol, which caused a considerable change both in the surface resistance to dyeing and in the ease with which the dye molecules penetrate into the fibre proper. In fact, the ease of admission through the surface skin increased six-fold, and the ease of penetration into the fibre increased 20-30 times. In both cases dyeing took place from a water bath without addition of solvent.

It seems from this simple experiment that the alcohol must remove something from both the surface and the interior of the fibre so as to provide greater ease of penetration. One point worth noting is that the diffusion coefficient after extraction with alcohol— 1.2×10^{-10} cm.² min.⁻¹—is almost exactly the same as for the diffusion of the dye into horn keratin. It may well be that ethanol, which we know can penetrate wool quite readily, removes some wool grease or fatty acid from the surface and probably from the interior of the fibre to make it more akin to horn keratin, which has not been exposed to wool grease as have the fibres.

Butyl alcohol (butanol) penetrates only very slowly into dry wool, but it is rapidly absorbed from aqueous solutions; under those conditions it might be expected to extract material from wool in the same way that ethanol does. However, unless the butanol-treated wool is later extracted with diethyl ether, material removed from the interior of the fibres by the butanol remains deposited at the fibre surface, insulating it from the dye. This can be seen from the reduced value of the surface admittance in Experiment 6 as compared with that in Experiment 2. No value is given for the diffusion coefficient in Experiment 6, because the surface resistance of the fibre entirely controlled the dyeing rate. Clearly, the diffusion coefficient for dyes inside the fibre had been much increased.

The most interesting results are those from Experiment 5, where solvent-dyeing conditions obtained, together with removal of the contaminant from the surface of the fibre by ether extraction. Here there is great ease of admission of the dye through the skin; in fact, the dye passes through the skin twenty times more readily than it did through the skin of the untreated wool fibre, and once through the skin it diffuses into the fibre at a rate about seventy times that for the untreated material. These ratios seem ample to explain the improved rates of dyeing that can be obtained with butanol in the dyebath when operating at low temperatures, for these experiments were all done at 30°C. Indeed, dyeing may become so fast that levelness may be difficult to obtain in piece dyeing, but there does seem to be a possible outlet in dyeing of loose wool, where levelness is not as important, and where a standing bath can be used, so that loss of butanol is not unduly costly. But apart from the practical aspect of the use of butanol, there is the further fascination for the academic worker of deciding how the presence of butanol inside the fibre can speed up diffusion so much. There is, obviously, wool grease or some such foreign matter in the so-called "clean" fibre, which retards diffusion. Then there is the true wool fibre, or what we like to call the true wool fibre after ethanol extraction; finally, by putting in another foreign substance (butanol) the rate of diffusion in the fibre can be increased.

These are all surprising phenomena, and it is even more surprising that wool dyeing can be so profoundly affected by the influence of solvents, soaps, greases, and so on. Admittedly, the results that I have given were obtained at 30°C. and many difficulties can be avoided by dyeing at the boil, but nevertheless one may wonder how such excellent results are obtained by the practical dyer. No doubt unevenness in individual fibres gives a lift or a sparkle to the appearance of a fabric; indeed, I always think that *mélanged* tops give a fabric a dull appearance compared with one made from dyed and undyed fibres. None the less, this sensitivity of wool to extraneous factors can cause real difficulty; we are all well aware, for example, of the results that may arise when batches of yarns of different age are mixed in one length of fabric that is to be piece-dyed.

It is fairly obvious from recent work of the Wool Industries Research Association that some of these dyeing problems have their origins in phenomena such as those that I have just described. Indeed, effects due to wool grease and fatty acids as contaminants on the surface or in the fibre can account in some measure for the difficulties that arise when dyeing wool from different sources, or wool that has been stored for different times. For example,

a dry-combed top was extracted for 10 min. with petroleum ether. It was then dyed with Naphthalene Orange G (C.I. Acid Orange 7), for 30 min. at 35°C. The extreme unevenness of dyeing was very obvious, very heavy dyeing occurring at the tip ends, while the root ends were scarcely touched. Next, fibres from the same top were dyed after having been cleaned with aqueous butanol; the dyeing was much more even—it seems that the solvent deposited a fairly uniform layer of hydrophobic material on the fibre surfaces and that this controlled the dyeing rate. When the root ends of pen-grown merino fibres, which had been thoroughly extracted with aqueous solutions of propanol and butanol and then dried and rinsed with petroleum ether, were dyed with Naphthalene Orange G for 15 min. at 35°C., a very even dyeing was obtained.

Finally I must mention the interesting phenomenon of the different dyeing behaviour of ortho- and para-cortex. I do not wish to attempt any explanation of this or other phenomena or to suggest how any unevenness they give rise to may be overcome—if, indeed, we do want to overcome them. They are of interest to the physicist and the chemist, and when the mechanics of their origins have been fully discovered I have little doubt that this knowledge will help to promote improved dyeing—either more uniform dyeing of large areas of fabric or more regular dyeing of individual fibres.

* * *

Wool is a wonderful fibre with a long list of virtues for the dyer and finisher. Indeed, as I come to the end of this lecture, I realise how rash I was when accepting the title "Wool as a Fibre in Dyeing and Finishing". I have merely touched on two features of the fibre, neglecting such powerful effects as felting and all the chemical wonders of this sensitive protein molecule. It is a fibre of great beauty and versatility and must have appealed to one with the qualities of George Douglas. He was a man of versatility and must have enjoyed greatly the beautiful fabrics that were produced under his guidance. I deeply appreciate the honour of presenting this lecture today as a tribute to his memory.

In conclusion, may I thank my colleagues at Torridon for assembling much of the material for the lecture, and in particular Mr. Medley, who has enlightened me on the power of solvents as an aid to diffusion.

WOOL INDUSTRIES RESEARCH ASSOCIATION
TORRIDON
HEADINGLEY
LEEDS 6

(MS. received 13th October 1960)

COMMUNICATION

An Autoradiographic Method Based on Tritium for Locating Resin Finish in Textiles

G. S. PARK

The preparation of tritium-labelled formaldehyde and its use for the autoradiographic determination of the distribution of urea-formaldehyde resin finish in textiles are described. The resin is found to be uniformly distributed in any filament cross-section with no resin layer on the surfaces, except in certain unusual cases. The distribution of resin across fabric sections is consistent with migration occurring during drying to those regions where drying is the most rapid. The results of some unusual methods of treatment are given. Resin distribution in a blended fabric has been determined. The autoradiographic method and a staining method for the determination of resin distribution have been compared.

INTRODUCTION

The application of resin-finishing treatments to cellulosic fibres has recently become more common. Resins based on formaldehyde, such as the urea-formaldehyde condensate, have been used to produce stiffer crease-resistant cotton and rayon fabrics with lower water imbibition. Uniform distribution of the resin throughout the fabric is probably required to produce the best properties. High resin concentrations just inside the filament surfaces are likely to give peculiar fibre properties, and interfibrillar adhesion can be caused by resin deposited on the filament surfaces. An unambiguous determination of the distribution of resin through fabrics and in filaments is therefore of considerable importance.

Previously two main techniques have been used for such investigations, one depending on the lowering by the resin of the accessibility of the filament to staining agents¹ and the other revealing the presence of resin by chemical reaction², or by interaction with a dye substantive to the resin³. The accessibility method has given useful qualitative information on fabrics, but is not as successful when applied to single filaments. Furthermore, the accessibility is controlled by the degree of polymerisation of the resin as well as by the amount present¹ and so gives a complex measure of the degree of resinification. Resin deposited on filament surfaces is not detected by this method, but it is likely to produce a barrier to diffusion into the filament and so give a faulty indication of the resin content. Chemical and selective dyeing methods depend on the chemical nature of the resin, which may be modified by differing degrees of cross-bonding. The results may also be modified by changes in accessibility to the reagents used. Because of these disadvantages we decided to use an autoradiographic technique on fibres containing radioactive resin to determine the evenness of resin distribution in individual filaments.

The resin is located by the effect on a photographic emulsion of the nuclear particles emitted from the decaying atoms, so that the problems of differing accessibility and differing chemical reactivity are avoided. Such a method, using ¹⁴C as the radioactive source, has been developed by Enders^{4,5} and his co-workers. We have used a modified method in which tritium (a radioactive isotope of hydrogen) is used to improve the

resolution given by the autoradiographs. Briefly, the autoradiographs are prepared as follows. A specimen of fabric containing tritium-labelled radioactive resin and set in a mounting medium is cut into sections. Squares of autoradiographic emulsion are coated on to the section surfaces and left to form a latent image which is later developed to reveal the location of the radioactive sites.

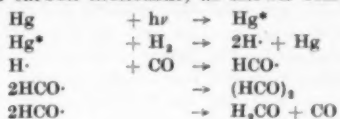
CHOICE OF RADIOISOTOPE

Of the atoms contained in the normal formaldehyde-based resins used in textile finishing, the radioisotopes of nitrogen and oxygen are too short-lived to be useful. The most useful isotopes are those of carbon and hydrogen of mass 14 and 3, respectively. Both of these emit β -particles (electrons) as they decay, with energies of 155 and 18 keV, respectively. Because of the energy difference, the penetrating power of β -particles from ¹⁴C is much greater than that of β -particles from tritium (³H). This has two consequences, the first being concerned with the escape of β -particles into the emulsion layer. This can be affected by the decay of ¹⁴C nuclei at a depth of 30 μ . or more below the surface of the cut section, whereas tritium at a depth greater than 1 or 2 μ . produces no photographic action. The second effect is concerned with the degree of penetration into the emulsion layer. A β -particle source on the emulsion surface will produce a hemisphere of latent image with radius proportional to the penetrating power of the particles. β -Particles from ¹⁴C would be expected to produce a sphere of radius more than 10 times that produced by tritium β -particles. Both these effects lower the resolving power of autoradiographs made with ¹⁴C and, since results^{4,5} suggest that a technique based on ¹⁴C is not good enough to establish the precise distribution of resin within an individual filament, we decided to use tritium.

The usefulness of tritium as a labelling atom in autoradiographic studies is limited by the labile character of the hydrogen linkage. In urea-formaldehyde and melamine-formaldehyde resins only the hydrogens in the formaldehyde residues are non-ionic and hence in stable combination with the resin; a urea-formaldehyde resin with some of the hydrogen of the formaldehyde replaced with tritium was therefore made for the present studies. The first stage in the preparation was the synthesis of radioactive formaldehyde (a mixture of formaldehyde and tritioformaldehyde).

SYNTHESIS OF RADIOACTIVE FORMALDEHYDE

Synthesis was achieved photochemically from radioactive hydrogen and carbon monoxide by circulating the mixed gases saturated with mercury vapour through a quartz tube illuminated with mercury resonance radiation⁶. Some glyoxal is produced under these conditions, but the major product is radioactive formaldehyde. The reaction is thought to proceed by the intermediate formation of HCO (and ³HCO) radicals, which combine to produce glyoxal or disproportionate to formaldehyde and carbon monoxide, as shown below.



To carry out a preparation the system (M, L, Q and S) was evacuated and equimolar quantities of radioactive hydrogen and of carbon monoxide were transferred by the Toepler pump P into the reaction line to give a total pressure of about 40 cm. of mercury. The mixed gases were circulated through the mercury saturator S, the quartz tube Q and the trap L by automatically raising and lowering the reservoir R of the mercury-circulating pump M. L was immersed in liquid oxygen, the mercury vapour lamp V turned on, and circulation continued until about 20% of the gases had reacted. The U.V. source was extinguished and the permanent gases were pumped into W. The yield of radioactive formaldehyde was found by allowing L to warm up and measuring the pressure in the known volume of the circulating system. An

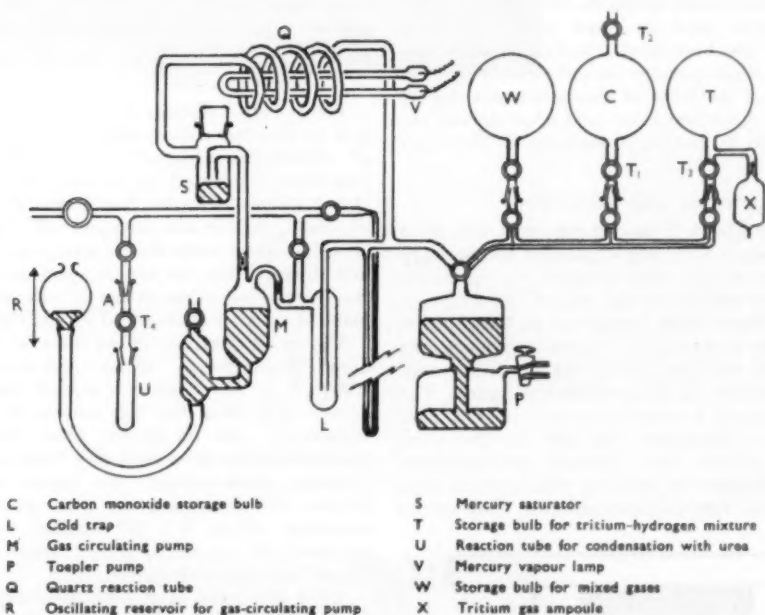


FIG. 1.—Apparatus for synthesis of tritioformaldehyde and condensation with urea

The apparatus used for the synthesis is shown in Fig. 1. The 500 c.c. bulbs C and T were used to store the gases. Pure carbon monoxide was generated in a separate system from formic acid by the action of sulphuric acid. It was dried and purified from sulphur dioxide by passing it through a cold trap at the temperature of solid CO₂. It was passed through C until all the air was displaced, the taps T₁ and T₂ were closed, and the bulb was transferred to the synthesising apparatus. One curie of pure tritium gas (0.41 c.c. at N.T.P.) was obtained from Harwell in a tube X with a break seal. This was joined on to the bulb T and, after the bulb was evacuated, the seal was broken with T₃ closed. Dried cylinder hydrogen was then allowed to expand from a large bulb into T to give approximately 500 c.c. of hydrogen-tritium mixture at N.T.P.

allowance of about 10% was made for the glyoxal in the product. In an average run a 30% yield (about 10 mg.) was obtained, the rest of the formaldehyde being lost as polymer on the glass and quartz surfaces.

PREPARATION OF LABELLED DIMETHYLOUREA AND TREATMENT OF FABRIC

The formaldehyde produced had a specific activity of ca. 50 curies/mole; this is at least ten times that required for autoradiography, so that in the preparation of dimethylolurea it was diluted with inactive formaldehyde.

Urea (42 g.) was dissolved in formaldehyde (90 c.c. of 37% soln.) and water (10 c.c.), warmed to 20°C. and the pH adjusted to 8.9–9.0 with sodium hydroxide. This is a normal formulation for a dimethylolurea precondensate with 10% of the

formaldehyde solution replaced by water; 100 mg. of labelled formaldehyde gas are therefore needed for each 2.7 c.c. of the solution to complete the precondensate mixture. As the yield of active formaldehyde was known, the volume of urea-formaldehyde mixture needed to make the complete precondensate could be calculated. The required volume was pipetted into the 10 c.c. tube U, frozen and outgassed. The trap L was surrounded by solid CO_2 to retain glyoxal and the formaldehyde distilled into U by cooling U in liquid oxygen. With T_4 closed, U and A were withdrawn. To form dimethylolurea U was heated to 80°C . in a beaker of water for 20 min. and allowed to cool for 1 hr. This simulated on a small scale the normal large-scale precondensation procedure. Each 3 c.c. of precondensate solution were diluted before use with 7 c.c. of 0.385% ammonium thiocyanate solution. Although aqueous thiocyanate was used in most cases, alcoholic solutions have also been used. The final mixture was used to treat slightly more than its own weight of cloth. Various methods of treatment and drying were used, as described later, and after drying the resin was cured by baking in an oven at 135°C . for 6 min.

MOUNTING AND SECTIONING

A rectangle about 2 cm. \times 1 cm. was cut from the treated fabric with edges parallel to the warp and weft yarns. It was gripped by pinching sectioned brass strips over the ends of the rectangle (Fig. 2), one brass strip being two to three times as wide as the rectangle to act as a support, the other being of the same width as the fabric and acting as a weight, as is shown in the figure. The fabric was hung in a small glass tube ($2\frac{1}{2}$ cm. long and $1\frac{1}{4}$ cm. in diameter) and left in the oven at 45°C . for about 1 hr. Methyl methacrylate, freed from inhibitor by shaking with caustic soda and then dried over calcium chloride, was mixed

with a chlorinated benzoyl peroxide paste Lupercol CDB ($\frac{1}{2}\%$). Polymerisation was started by heating over a Bunsen burner; when the liquid had become highly viscous the tube containing the specimen was filled and kept at 45°C . overnight. The glass tube was broken away from the polymethyl methacrylate block containing the fabric.

When ^{14}C is used to label resin-treated specimens, the β -particles are so penetrating that it is necessary to cut very thin sections if autoradiographs of good resolution are required. This is not necessary with tritium-labelled specimens and so it was possible to use quite thick slabs which could be polished to give an extremely smooth surface. The block containing the fabric was mounted in a jig and sawn into discs 1-mm. thick, using a stream of water to keep the block cool and to carry the radioactive fragments into a large beaker. The discs were polished on glass plates, using four grades of polishing alumina and finishing off on a piece of chamois leather.

PREPARATION OF AUTORADIOGRAPH

The polished section was attached with Durafix to a microscope slide coated with a very thin layer of chromium-hardened gelatin. Following the procedure suggested by Kodak⁷, a small amount (1 or 2 sq.cm.) of Kodak fine-grain autoradiographic emulsion, AR10, was stripped off a plate, floated on clean water with the sensitive face downwards, and draped over the active specimen by bringing the microscope slide under it. It was dried in a blast of cold air and stored in the dark.

The specific activity of the fabric in the block was about 20 meuries/g. If the resin is uniformly distributed in the filaments and if the β -particles escape only from the top micron of the polished section, it can be shown that about 3×10^9 particles are emitted each day from each sq.cm. of filament cross-section. To cause a reasonable degree of blackening in the autoradiographic emulsion, about 5×10^9 tritium β -particles are required per sq.cm. of film⁸; hence exposure for about two days is required.

After the lapse of at least two days, the latent image was developed in Kodak D19b developer, fixed in a hardening fixing bath and washed. After being detached from the section, the processed emulsion was attached under water to another gelatin-covered slide, dried and examined under the microscope. Separation of the autoradiograph from the section (which is made much easier by using a polished section instead of a microtomed one) enabled the two to be examined separately and so avoided confusion. Dark regions in the autoradiograph must be due to a high formaldehyde content and not to optical effects in the cross-section. The polished section can be examined under the microscope by vertical illumination and compared with the autoradiograph.

The resolution obtainable in autoradiography depends on the grain size of the photographic image, as well as on the penetrating power of the radiation from the isotope. Fig. 3 gives an indication of the resolving power of our system. A very thin layer of resin was deposited on filament surfaces from contaminated squeeze rollers; a

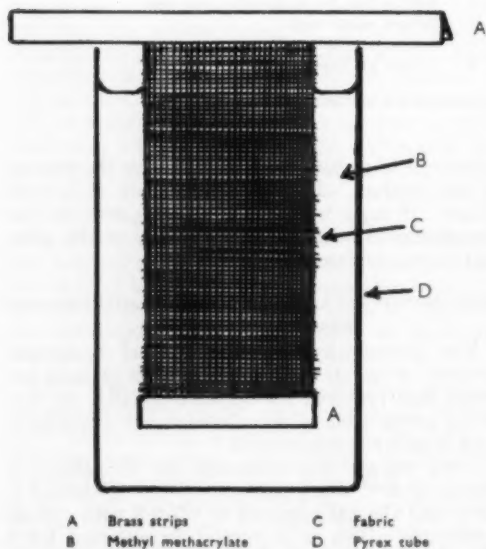


FIG. 2—Mounted fabric

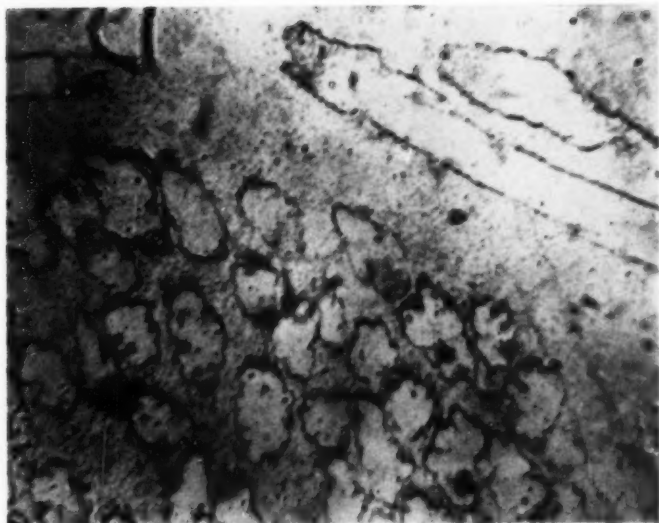


FIG. 3—Autoradiograph of cross-section of viscose rayon fabric showing surface resin applied from squeeze rollers ($\times 400$)

FIG. 4—Autoradiograph of cross-section of resin-finished viscose rayon fabric ($\times 50$)

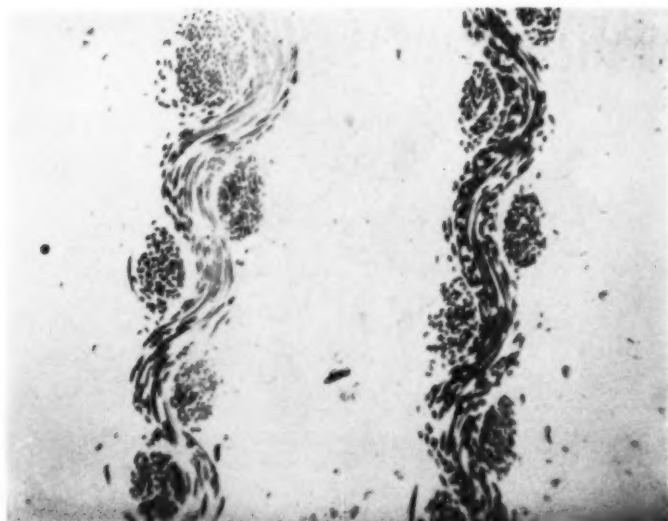
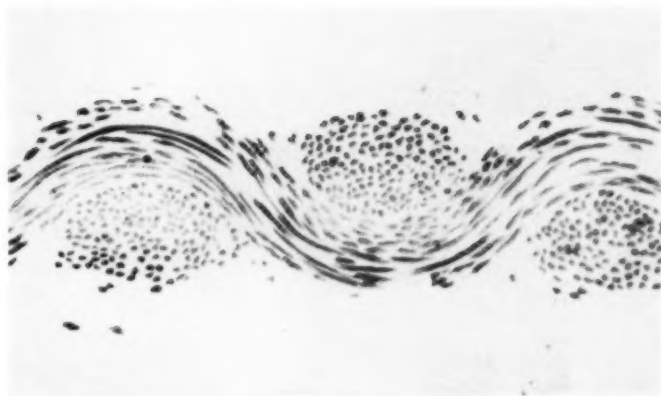


FIG. 7—Autoradiograph of cross-section of resin-finished double-layered viscose rayon fabric. Heated surface is on the right ($\times 50$)



FIG. 5—Autoradiograph of cross-section of resin-finished viscose rayon filaments ($\times 400$)



FIG. 6—Cross-section of resin-finished viscose rayon filaments ($\times 400$)

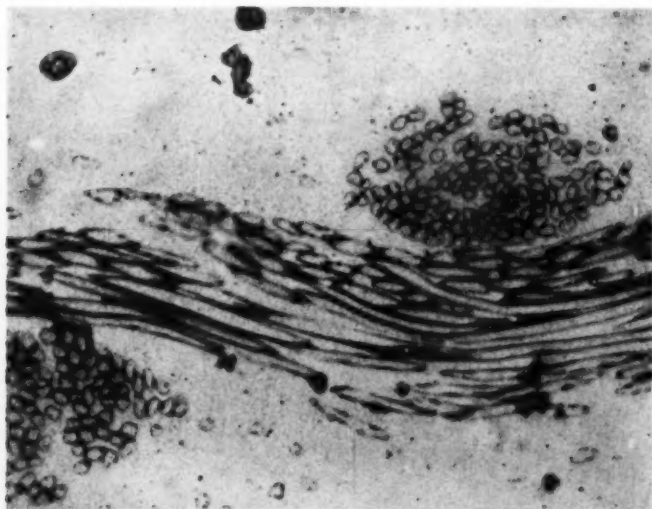


FIG. 8—Autoradiograph of cross-section of viscose rayon fabric resin-finished with alcoholic solution ($\times 100$)



FIG. 9—Autoradiograph of cross-section of double-layered viscose rayon fabric resin-finished with alcoholic solution. Heated surface is on the right ($\times 100$)



FIG. 10—Autoradiograph showing surface resin cementing viscose rayon filaments together ($\times 400$)

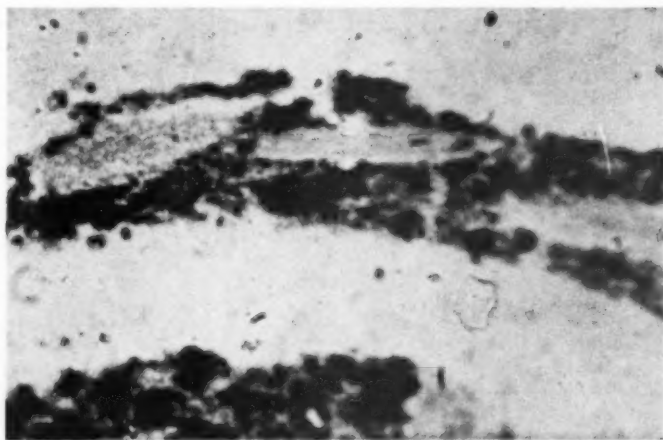


FIG. 11—Autoradiograph of surface resin from highly condensed resin precondensate ($\times 400$)

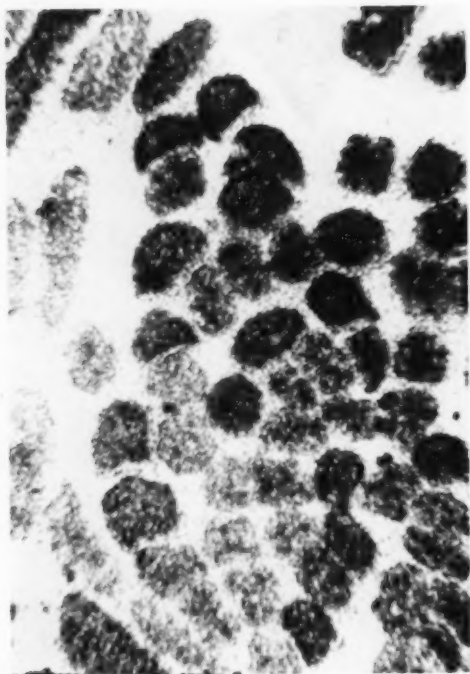


FIG. 12—Autoradiograph of cross-section of resin-finished Fibro-Fibrolane blend ($\times 400$)

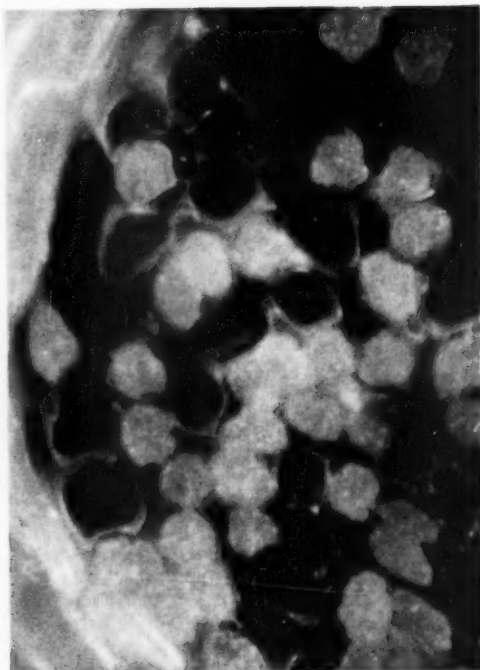


FIG. 13—Cross-section of resin-finished Fibro-Fibrolane blend. Fibro filaments appear light and Fibrolane ones dark ($\times 400$)



FIG. 14—Autoradiograph of the stained section ($\times 50$)

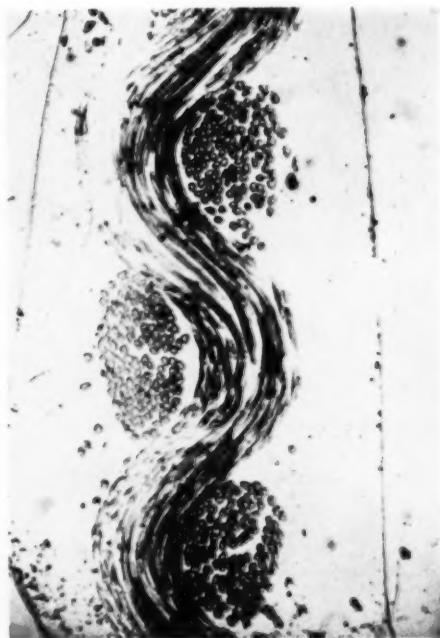


FIG. 15—Microtomed section of resin-finished and stained viscose rayon fabric ($\times 50$)

photomicrograph of the autoradiograph prepared from the filaments is shown in the plate. The resolution appears to be about 1 or 2 μ .

STABILITY OF TRITIUM-CARBON BOND

Contrary to an earlier report by Klar⁹, it has been shown by Wirtz and Bonhoeffer¹⁰ that, even after prolonged standing in presence of acid or alkaline catalysts, no exchange of hydrogen occurs between formaldehyde and heavy water. This has been confirmed by Walters¹¹, and so it is to be expected that the carbon-tritium bonds in formaldehyde, dimethylolurea and urea-formaldehyde resin will be stable. Nevertheless, two kinds of test have been used to show that the autoradiographs are not influenced by tritium migration. If tritium exchange occurred in the production of dimethylolurea, tritiated water would be formed and could exchange with the hydrogen on the cellulose hydroxyl groups. Scouring of the resin-finished fabric would then be expected to remove the tritium and alter the character of the autoradiograph obtained. Autoradiographs of scoured and unscoured fabrics have been compared, and though the latter are less intense than the former the general character of tritium distribution is not changed. It is well known that a mild scour removes some of the resin from a urea-formaldehyde-finished fabric; some decrease in intensity is therefore to be expected and it is probable that none of it is due to tritium exchange. Another approach to the problem is by direct treatment with tritiated water. Autoradiographs have been made of fabric treated with a normal non-radioactive dimethylolurea solution prepared in tritiated water. With fabric dried immediately after treatment no autoradiographic image was found; a faint image was, however, produced when the fabric was left for a period before drying. It appears therefore that, even if exchange could take place between water and formaldehyde, the tritium atoms exchanged would be unlikely to affect the autoradiograph appreciably if the normal resin-finishing technique were used.

RESULTS OBTAINED BY NORMAL RESIN TREATMENTS

A strip of plain-weave 4½-den. spun-rayon cloth was soaked in a mixture of radioactive precondensate and aqueous catalyst. It was passed twice through rubber rollers to remove excess solution and dried at about 70°C. by streams of air directed at the fabric faces from two symmetrically placed hair dryers. The fabric was cured, mounted and sectioned.

A photomicrograph of an autoradiograph is shown in Fig. 4. The darkening in the autoradiograph is a measure of the resin content of the cross-section. There are two noticeable features. In the first place the fabric surfaces have produced more darkening than the inner regions; in particular it appears that the cross-over points between warp and weft threads are deficient in resin. A result similar to this has been reported by Enders⁴, and staining experiments have shown either a low resin content or a less polymerised resin at the warp-weft cross-over points¹². The

low resin content probably results from a migration of the resin precondensate to the drying surfaces from the regions shielded from the drying air draught. A similar phenomenon is observed on casting polymer films from solution. The greatest thickness of polymer occurs in the region where the solvent evaporation is most rapid. The second feature concerns the resin distribution in individual filaments. The resin penetrates the whole filament and is not limited to a ring in the outer regions.

The photomicrographs at higher magnification in Fig. 5 and 6 illustrate this somewhat better. Fig. 5 shows the autoradiograph of the oblique filament cross-sections shown in Fig. 6. The correspondence between the two photomicrographs is further indication of the resolution obtained in this kind of autoradiography. The uniformity of blackening of the autoradiograph across any one filament section shows the uniformity of resin concentration. In view of the variation of resin concentration between one filament and another, the uniformity across the filament section is striking.

It might be thought that uniformity is produced by a uniform smearing of radioactive powder over the filament cross-section during the polishing process. If, however, finished and unfinished fabrics are mounted side by side in the same block and polished together, no activity is found to be transferred to the unfinished cross-section or to the mounting medium. Other evidence against this hypothesis is provided by the existence of the non-uniform autoradiographs of filament cross-sections discussed below.

EFFECT OF UNEVEN DRYING

If the resin distribution indicated by Fig. 4 is due to migration from the warp-weft cross-over regions to parts of the fabric where more rapid drying occurs, it might be thought that fabric dried from one face only would show a preponderance of the resin on that face. Autoradiographs of a single layer of fabric do not show any marked differences between the dried and undried faces. A special double fabric consisting of two layers with a common warp thread each centimetre has, however, enabled us to demonstrate very clearly the effect of uneven drying. This fabric had been used by D. Wilson of Courtaulds Ltd. Bocking laboratories to show drying migration on a macroscopic scale and we are indebted to him for a small sample. A strip of the fabric, after being treated with radioactive dimethylolurea, was dried by wrapping it round a heated brass cylinder (at 100°C.). Fig. 7 shows an autoradiograph of the cured fabric. The difference between the layer of the fabric in contact with the hot surface and the outer layer (shown on the left of the figure) is very marked and provides striking confirmation of the migration of the precondensate to the heated surface. The lowest resin concentration is found on the inner face of the outer layer. This is not surprising, since some drying will be occurring on the outer face of this layer, so that a somewhat higher concentration of resin will be produced there. The absence of any noticeable effect on the single-layer fabric may be due to the open nature of the material, which

ensures that both faces of the fabric dry at about the same rate.

TREATMENT IN ALCOHOLIC SOLUTION

Fabric that has been padded with an alcoholic solution of dimethylolurea, dried, and heated to the curing temperature has a stiff cambric-like feel quite unlike a normal resin-finished fabric. This suggests that the resin is on the outside of the filaments, cementing them together and stiffening the whole fabric. A chemical method for locating resin finish using Tollens reagent indicated that dimethylolethyleneurea resin applied from alcoholic solution only just penetrates the surfaces of the filament, though when the resin is applied from aqueous solution uniform penetration into the filament is obtained¹³. By selectively dyeing the resin with Anthraquinone Blue BN (C.I. Acid Blue 45), Monroe¹⁴ has demonstrated a similar effect for dimethylolmelamine resin. Using a different dye, Duranol Violet 2R (C.I. Disperse Violet 1), Beaumont¹² has shown, on the other hand, the presence of resin particles in the small capillaries between the filaments after application of dimethylolurea by spraying or from alcoholic solution. Fig. 8, an autoradiograph of a fabric finished with tritiated urea-formaldehyde resin applied from alcoholic solution, shows the deposition of most of the resin outside the filaments. A close examination of the plate shows that many of the filaments are pinned together at contact points with the resin, and in the warp-weft cross-over region the fabric is stiffened by the cementing of the warp threads to the weft. Fig. 8 indicates that very little resin penetrates into the filaments, but experiments on the double fabric mentioned in the previous section and using the drying technique outlined there indicate that penetration depends on the rate of drying. An autoradiograph from the layer of the double fabric that was dried slowly after treatment in alcoholic solution is shown in Fig. 9 on the left. The layer on the right was dried rapidly in contact with the hot surface. This shows that more penetration into the filaments occurs when the drying rate is slower; in consequence less resin is left in the interstices to cement the filaments together. The resin that penetrates into a filament is not uniformly distributed, but occurs mostly near the filament surface.

Surface resin is sometimes observed between parallel viscose rayon filaments even when the dimethylolurea has been applied in aqueous solution. Fig. 10 shows an example in which a layer of resin is cementing together two viscose rayon filaments. In this example the filaments had been fully swollen with water before treatment with the resin. In the large-scale resin finishing of cellulose that has never been dried, inter-filament adhesion is found if insufficient squeeze pressure is used for driving the water out of the filaments; this was suspected to be caused by surface resin, and Fig. 10 confirms this possibility.

Another example of inter-filament resin is shown in Fig. 11. This is an autoradiograph of the oblique section of three filaments in a viscose rayon

fabric that has been treated with an aqueous resin precondensate and left moist for several days before drying and curing. The waiting period allows further condensation to occur on the filament surfaces, and the molecules thus formed are too large to penetrate on subsequent drying and curing. As is shown in the plate, this can result in the formation of rather large resin particles between the filaments.

These examples of surface resin are the exception rather than the rule and occur only to a very minor extent in normal resin finishing.

RESIN DISTRIBUTION IN BLENDS

When a resin finish is given to a fabric consisting of two or more different kinds of filament, it is likely that different resin contents will be obtained in the different filaments. Owing to the different affinities of the different fibres, staining techniques fail completely in such cases, but autoradiography offers a simple and direct method of determining resin distribution. Fig. 12 shows a portion of the autoradiograph of a resin-finished fabric made from a Fibro-Fibrolane blend, and Fig. 13 is a photomicrograph taken by vertical illumination of the corresponding portion of the cross-section. Since the Fibro contains titanium oxide as a delustering agent and the Fibrolane does not, the former can be recognised by its light appearance in Fig. 13. The Fibrolane filaments, on the other hand, appear darker than the background. A comparison of the two plates shows that in most cases the Fibrolane filaments give rise to darker regions on the autoradiograph than the Fibro filaments in the immediate vicinity. Although darker regions are formed by Fibro filaments in the outer parts of the fabric than by Fibrolane filaments in the inner region, this is due to the general distribution of resin illustrated by Fig. 4 and the over-all picture is one of greater radioactivity in the Fibrolane filaments. It might be argued that, because of the different β -particle absorptive powers, the intensities of the autoradiographic images from Fibro and Fibrolane could not be used as a measure of the tritium content. To a first approximation, however, the β -particle absorptive power is the same for all materials if the thickness used is inversely proportional to the density. A consequence of this is that, even for media of different densities, the β -particle emission is still proportional to the radioisotope concentration per unit mass of the medium. Fig. 12 can therefore be taken as showing a higher resin content in the Fibrolane than in the Fibro. Other portions of the autoradiograph do not show such a large difference between the two kinds of filament; autoradiography, unlike a macroscopic analytical method, enables these differences in different parts of the fabric to be detected.

CORRELATION WITH THE STAINING TECHNIQUE

The autoradiographic method for determining the distribution of resin finish, though unambiguous, is not as convenient as the staining method, which can be used on any piece of cloth that has been resin-finished in the factory or the laboratory. The autoradiographic method, on the other hand,

is restricted to laboratory treatment on a very small scale, with all the usual precautions needed in radioactive experimentation. In consequence it is helpful to know to what extent the resin distribution obtained by the staining technique represents the true resin distribution given by autoradiography. It has been demonstrated that, at constant resin content, the greater the degree of condensation of the resin the weaker is the stain produced. Nevertheless it appears likely that in any one small region of finished fabric the variation in degree of condensation might well be quite small and the degree of staining could then be taken as a measure of the amount of resin present.

To test this hypothesis an uneven radioactive resin finish was given to a small area of fabric. The fabric was stained with a combination of Brenthol CT (C.I. Azoic Coupling Component 8) and Brentamine Fast Red 3GL Salt (C.I. Azoic Diazo Component 9)¹ and mounted in polymethyl methacrylate. The polished sections used in all our previous work were too thick to enable a good estimate of stain intensity to be obtained; microtomed sections were therefore prepared. The sections were stuck to the microscope slides and used to make autoradiographs. After the processed autoradiographs had been floated off, the sections were mounted in Canada balsam for microscopic examination. Fig. 14 is the autoradiograph of the section shown in Fig. 15. Although we have not been able to produce by this method autoradiographs that are as good as those from polished sections, it will readily be seen that the dark regions on the autoradiograph correspond to the light regions on the section and *vice versa*. We

therefore conclude that the stain distribution can be used as a reasonable qualitative guide to the resin distribution within one piece of cloth in which all the resin is cured for the same time at the same temperature.

* * *

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CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Wet Crease-recovery

Since about 1954 there has been a growing realisation of the importance of crease-recovery of wet fabrics in connection with their smooth-drying characteristics; furthermore, there has been renewed interest since 1957 in the old observation that certain treatments may produce a fabric which possesses wet recovery but not dry recovery.

It is obviously desirable to have some objective method of measuring wet recovery; a modification of the Monsanto method was suggested four years ago¹ and seems to have been generally accepted in the U.S.A. According to these results, however, the recovery of bleached and untreated cotton fabric is 165° when dry and 176° when wet. Further results obtained some three years later by the same test procedure show the following comparisons²—

	Crease Recovery (°)	
	Dry	Wet
Bleached broadcloth	178	168
Mercerised broadcloth	151	171
Mercerised print cloth	197	203
Bleached broadcloth	165	176

Additional results published this Spring³ include the following figures—

	Crease Recovery (°)	
	Dry	Wet
Bleached print cloth	200	194
Mercerised print cloth	192	217

Lest it should be thought that some of these rather remarkable results are due to the practice of expressing the angular recovery as the sum of warp and weft recovery figures, it is interesting to consider some very recent measurements where warp and weft recoveries are expressed separately⁴—

	Crease Recovery (°)			
	Warp		Weft	
	Dry	Wet	Dry	Wet
Bleached cotton print	89	85	91	104
Bleached cotton print	91	93	84	94
Bleached cotton print	79	64	80	82

It is submitted that these figures are not in accordance with reality. If one applies the "clenched-fist" test to untreated cotton fabrics, i.e. fabrics which have not been given the crease-resist finish, the dry fabric will recover to a modest but appreciable extent, whereas the wet fabric often shows little or no recovery and remains compressed. The difference between the two is remarkable.

If, however, the wet crushed sample is gently opened by hand and compared with the dry crushed sample, it will be seen to contain very many more creases, but these are less sharply defined than those in the dry fabric. Nevertheless, a wet untreated cotton fabric, when crushed, remains crushed to a much greater extent than a similar fabric crushed in the dry state; this is contrary to the general run of results from the Monsanto test.

It is suggested that the anomaly is largely due to the operation of the Monsanto apparatus, whereby one end of the creased strip is allowed to hang vertically and the other end is held in a clamp which is adjusted from time to time around a protractor device by the operator so that the free end is moved to coincide with a vertical line; in other words, the Monsanto method is an assisted recovery test.

As might be expected, a cellulose fabric in the wet and plastic state is very sensitive to assistance, the application of which can provide unrealistic results.

It may be that a correct picture is given by this method of test when applied to a series of treated fabrics to measure dry recovery, or to another series of differently treated fabrics to estimate wet recovery, but what reliance should be placed on it

as a test for dry and wet recovery in the same sample, when it places the untreated materials in the wrong order?

There are tests of recovery from creasing which may be described as unassisted, i.e. they rely on natural recovery, and these place the measurements of dry and wet recovery in untreated fabrics of both cotton and rayon in the right order.

Quite apart from the matter of assisted or unassisted tests of recovery from creasing, a correct interpretation of angular recovery in the wet state as compared with the dry state is surrounded by many difficulties when laboratory tests are employed on an objective basis. It is not easy to evaluate the behaviour of a garment in use by testing one rather small crease amongst very many in the wet fabric, and one large sharply defined crease amongst not so many in the dry fabric.

J. T. MARSH

TYSELEY
1 BOWER ROAD
HALE
CHESHIRE

20th August 1960

¹ Lawrence and Phillips, *Amer. Dyestuff Rep.*, **45**, 548 (1956).

² Guthrie, *Text. Research J.*, **29**, 834 (1959).

³ Reeves, Perkins, and Chance, *ibid.*, **30**, 179 (1960).

⁴ Reid, *et al.*, *ibid.*, **30**, 495 (1960).

Notes

Election of Fellows

At a meeting of Council held on 7th September 1960, the following were elected Fellows of the Society—

Robert C.ifford Cheetham

Audenshaw, Lancs; Wool Technologist and Research Chemist, Courtaulds Ltd.

Pavel Krug

Manchester; Manager of Textile Section, Hardman & Holden Ltd.

Dennis Michael Nunn

Bradford; Works Manager, Low Moor & Water Lane Ltd.

F. Malcolm Stevenson

Duffield, near Derby; Director and Manager, Stevenson (Dyers) Ltd., Ambergate

Bristol Symposium 1961

The Society will be holding a symposium in Bristol on 20th–22nd September 1961. The subject is "Science and Craft in both Textile and Non-textile Coloration".

The Technical Papers Subcommittee invites any members or subscribers who are carrying out work which they believe will be suitable for presentation at this symposium to submit a brief synopsis (not exceeding 200 words) before 14th January 1961. After synopses have been considered by the Subcommittee, invitations to prepare papers for inclusion in the pre-prints will be issued.

Synopses should be addressed to The Chairman, Bristol Symposium Technical Papers Subcommittee, c/o S.D.C., Dean House, 19 Piccadilly, Bradford 1, Yorkshire.

A.S.D.C. Examinations 1961

The examinations for the Associateship of the Society of Dyers and Colourists will be held on 1st, 2nd and 3rd June 1961. Application forms, which must be returned by 28th February, are available from the General Secretary.

Colour Index Additions and Amendments

Publication of additions and amendments to the *Colour Index* will continue quarterly in 1961. The subscription for the four issues will be £2. Further particulars may be obtained from the General Secretary.

International Symposium on Inorganic Polymers

A symposium sponsored by The Chemical Society will be held in the University of Nottingham on 18th–21st July 1961. The symposium will

consist of Main Lectures and Contributed Papers. Contributed papers will be divided into six sections, viz. polymers containing (1) transition metals, (2) a Group III element, (3) a Group IV element, (4) a Group V element, and (5) a Group VI element, and (6) other aspects, e.g. analytical methods. The main emphasis will be on the preparation and reactions of inorganic polymers, but consideration will also be given to such topics as thermal stability, and the mechanics and kinetics of condensation.

Full details will be available in March 1961 from the General Secretary, The Chemical Society, Burlington House, London W.1.

1961 Conference of the Oil and Colour Chemists' Association

The Biennial Conference of O.C.C.A. will be held at Torquay on 30th May-3rd June 1961. The general title will be "Physics in Surface Coatings". Further information is available from the General Secretary, O.C.C.A., Wax Chandlers' Hall, Gresham Street, London E.C.2.

Dyers Company of London

At a General Court held on 12th October 1960 for the election of Wardens for the ensuing year, Mr. A. C. M. Harding was elected Prime Warden and Mr. D. L. B. Marshall was elected Renter Warden.

Dyestuffs Office

Mr. H. Blackshaw, F.S.D.C., has been appointed to succeed Mr. Fred Scholefield, M.Sc., F.R.I.C., F.T.I., F.S.D.C., as adviser to the Dyestuffs Office and takes up his duties in November.

Mr. Blackshaw, a member of the Publications Committee, was a member of the *Colour Index* Panel and acted as Co-ordinator from 1947 to 1957. He was awarded the gold medal of the Society in 1958.

Meetings of Council and Committees October

Council—No meeting
Colour Index Editorial Board—5th
Finance and General Purposes—11th
Disperse Dyes Subcommittee—12th
Examinations Board—17th
Publications—18th
Fastness Tests Co-ordinating—18th
Textbooks and Monographs Subcommittee—18th
Review of Textile Progress—24th
International Relations—26th
Worshipful Company of Dyers Research Medal—28th
Diplomas—31st

OBITUARY NOTICE

John W. Hirst

John W. Hirst, who died in Australia on 14th April 1960, was born at Huddersfield on 25th June 1885. After leaving school at 13 he studied in the evenings at Huddersfield Technical College and was awarded a County Council Scholarship tenable at Leeds University, where he studied in 1908-9. After leaving the University he managed the dye-house of Geo. Hirst & Sons, Birstall, for two years, and in 1911 became dyer at Foy & Gibson Ltd.,

Australia, where he remained until his retirement in 1955.

Hirst was a member of the Society for over 50 years and was elected a Fellow in 1956. He was active in the foundation of the Australian branch, of which he was Foundation President; he was awarded life membership of the Society of Dyers and Colourists of Australia.

He is survived by his wife, daughter and son.

New Books and Publications

Plastics Progress 1959

Papers and Discussions at the International Plastics Convention 1959

Edited by Phillip Morgan. Pp. xii + 216. London: Iliffe & Sons Ltd. 1960. Price, 55s. 0d.

This book is a collection of papers read at the International Plastics Convention in 1959 and gives details of developments in some of the newer plastics, modifications of plastics, and plastics technology. Surveys of developments in glass-reinforced and expanded plastics are included. It will be of interest primarily to the plastics technologist, but the inclusion of fundamental theory and new experimental results in some of the papers provides material of interest to polymer chemists generally.

Two papers deal with polypropylene. The first, by J. M. Goppel, gives a useful classification and

discussion of different polypropylenes in relation to crystallinity and molecular weight, and a brief consideration of some aspects of moulding. Moulding of polypropylene is covered in more detail by G. Campbell, particularly such aspects as flow properties, specific volume, and crystalline structure in relation to moulding; moulding procedures are also discussed.

Three papers are concerned with developments in materials. H. N. Lewis discusses some new epoxide resins obtained by epoxidation of carbon-carbon double bonds by use of peracetic acid, and the flexibilising and hardening of these and other epoxide resins, with some applications. R. R. Smith considers cross-linking of thermoplastics after fabrication, by both irradiation and chemical methods, and gives results obtained using polyethylene, cellulose acetate and polyvinyl chloride.

J. Benton and C. M. Thomas describe some theoretical and experimental studies of block and graft copolymers and give a concise account of methods of preparation, a clear account of the theoretical principles involved, and details of some experimental results obtained using a duofunctional initiator.

Four short papers cover recent developments in glass-reinforced plastics. H. Tunteler considers their use and fabrication in the Benelux countries. R. Kraft deals with progress in Germany, and B. Parkyn considers U.K. developments in materials and moulding techniques and the performance of mouldings. The final paper in this group, by H. C. Blankmeyer, describes progress in the U.S.A., with particular reference to reinforcement, finishes, and dough and spray moulding.

Two papers are concerned with expanded plastics. B. L. Raskin defines and outlines methods of preparing foamed plastics and gives a short account of some commercial developments in the U.S.A., including the development of foamed plastic smokes and their applications. The other paper, by W. B. Brown, deals in some detail with bulk density and physical properties of expanded polystyrene.

Extrusion forms the subject of two papers. D. A. Lannon and G. C. Karas describe experimental studies of extrusion of thermoplastic materials and discuss the results in terms of viscosity, temperature, and other variables. L. Griffiths considers the extrusion of acrylics, discussing the extrusion behaviour of polymethyl methacrylate, extrusion equipment, and some extrusion products. This paper is well illustrated by photographs and diagrams.

The technical papers are well documented; not the least interesting parts of the book are the discussions following groups of related papers.

W. R. MOORE

Chemical Analysis

Volume XII

Systematic Analysis of Surface-active Agents

By Milton J. Rosen and Henry A. Goldsmith.

Pp. xvii + 422. London and New York: Interscience Publishers. 1960. Price, 102s. 0d.

The stated aim of the authors was to fill a gap in the analytical field by producing a comprehensive treatise on the analysis of surface-active agents, and in this they have well succeeded. Previously it was necessary to refer to the literature for up-to-date methods in this branch of applied science in which such rapid advances have been made in the last two decades.

It should be noted at the outset that the book is concerned exclusively with organic surface-active

agents and their salts. The merit of the book lies in the adoption of a classification system which is based on the elemental and functional groups present, so designed to meet the needs of the analyst. Thus the book should not be readily outdated as new surface-active agents become available, as they inevitably will.

The first chapter discusses the classification system adopted. This is followed by a series of tests for the detection of single surface-active agents, their isolation from other materials, and finally their quantitative estimation. Chapters 3 and 4 deal, respectively, with the functional qualitative and quantitative analysis of surface-active agents. Here mention is made of some of the newer analytical techniques such as X-ray diffraction, and infrared and ultraviolet absorption.

The final chapter discusses procedures for the separation of mixtures of surface-active agents. Preliminary information is obtained from ignition tests and elemental analysis, and a suitable method of separation is deduced from this. It is emphasised, however, that the purpose of separation of the mixtures is for qualitative identification of the components rather than fully quantitative analysis. This is disappointing, as many practising analytical chemists are concerned with quantitative analysis of the increasingly complex mixtures of surface-active agents available.

A comprehensive bibliography is given, but it would have been more convenient for the reader if the references had been given at the foot of each page rather than collected at the end of each chapter. With very few exceptions there are no references to the literature since 1957, which perhaps is surprising. Also, mention is made several times of recent developments, with references to the period 1948-53. A useful feature is the inclusion of *Chemical Abstracts* references to many of the papers.

Finally, there is an appendix giving tables of nominal chemical structure for commercial products, together with the composition and certain properties of some raw materials. It seems unnecessary in a book of this standing to include the names and addresses of the producers of surface-active agents; Table I is needlessly lengthened because of this. Similarly, the subject index could have been simplified somewhat, for example, by giving only one reference to Atlox instead of nine.

These criticisms are minor and in no way detract from the usefulness of the book, which is a "must" for all laboratories working in this field. Very few errors were noted, and the book is well produced and maintains the high standard set in this series.

H. L. WEBSTER

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952) and also, together with symbols and the periodicals abstracted, in the annual index

I—PLANT; MACHINERY; BUILDINGS

Machine for Printing Carpets

Melliand Textilber., 41 (Aug 1960) 996
A machine that prints carpets without crushing the pile. The pattern is mounted in relief on a roller and is covered by a sponge-like material that takes up the printing colour and releases it in the form of fine jets on being pressed against the carpet. W.M.

PATENTS

Pelleting of Carbon Black (C.I. Pigment Black 6 and 7)

Phillips Petroleum Co. USP 2,917,374 (26 Aug 1957)

Automatic Control of Tension during Winding or Batching of Textiles

Gebrüder Sucher BP 840,380 (Germany 31 Dec 1955)

Apparatus for Impregnating Carded Laps

Lantor BP 843,288 (9 Dec 1957)

Fluid Treatment of Beams of Fabric

Burlington Engng Co. BP 837,218 (2 Jan 1957)

A pressure-dyeing machine in which the liquor can be passed either way through cloth batched on a beam. It is very suitable for automatically controlled high-temperature dyeing. C.O.C.

Drying Cylinder

J. Dilworth BP 839,058 (3 Dec 1957)

A drying cylinder is formed of two concentric cylinders and is heated by steam passed from end to end through the space between the two cylinders. Condensation water formed on the inside of the outer cylinder can be removed by increasing the velocity of the steam. C.O.C.

Drying Apparatus for Use in Tenters or other Web-treating Machines

F. Sibel USP 2,916,796 (30 Aug 1955)

Jets for the hot air or other drying fluid are arranged so that the air travels in a straight path through the web and into the exhaust nozzles. This enables the apparatus to be used without a container and without contaminating the atmosphere of the room it is in. The jet nozzles are movable laterally, thus making the arrangement particularly useful for clip stenters, as their position can be adjusted whenever the rails have to be adjusted. C.O.C.

Automatic Control of Stenters or Weft Straightening Machines

A. Leimer BP 840,285 (Germany 2 Apr 1957)

A photosensitive device for counting the picks of the fabric as it is fed to an overfeed stenter or weft-straightening machine and its use in the automatic control of the processing. C.O.C.

Heating or Drying Sheet Material in Fluidised Beds

British Rayon Research Assocn. BP 838,784 (18 Feb 1956)

Weft tension is maintained in the sheet material as it passes through the bed by engaging its selvages by a pair of endless flexible members. Each of these members runs in and is supported by guideways, the clearance between the members and the guideways being such that the particles forming the bed cannot get between the moving parts. BP 838,785 (30 Nov 1956)

The container for the bed has at least one slit in a side wall for entry of the material to be treated. The width of the slit is such that the particles forming the bed cannot pass through it. C.O.C.

Screen-printing Machines

Reggiani Meccanotex BP 837,860 (Italy 21 May 1957)

A machine in which the number of manual operations to be performed during each cycle of operations is reduced to a minimum. BP 837,861 (Italy 21 May 1957)

Improved mechanism for the squeegee is described. C.O.C.

Drying or Conditioning Cloth, Paper or the like with a Gas

Aktiebolaget Svenska Fläktfabriken

BP 837,686 (Sweden 16 Nov 1957)

The cloth is treated with jets of the gas which impinge perpendicularly against its surface. The jets have small diameters and are evenly spaced over the surface to be treated. The gas is removed from the web by means of exhaust points placed between the jets. Thus the gas is forced to flow along the cloth from all directions radially toward the exhaust points from all the jets encircling such points. The compactness of the apparatus enables a very great quantity of gas to be used without increasing the running costs. C.O.C.

Machine for Applying Transfers Continuously to Woven and Knitted Fabrics

M. Levy USP 2,914,875 (U.S.A. 1 Mar 1956)

Napping Machine

I. Pasold BP 840,440 (23 July 1957)

A table has several oscillating, rough-faced plates. The cloth is passed between the faces of these plates and a fixed table close to them. The forces generated in the cloth by the movement of the individual plates more or less cancel one another. C.O.C.

Preventing Corrosion of Molten-metal Baths used in Dyeing (VIII p. 648)

Printing Screens (IX p. 648)

Water-repellency Testing—Effect of Shaking Specimens (XIV p. 656)

II—WATER AND EFFLUENTS

Clarification of Dyehouse Effluents by Magnesium Chloride

J. Nosek and J. Křepelka

Vodníhospodářství, 9 (1959) 556–557
Chem. Abs., 54 (25 June 1960) 12437

Full-scale tests have shown $MgCl_2$ to be a very effective clarification agent of highly alkaline effluents. Its use is especially advisable for laboratory tests, as the colour of the originally white $Mg(OH)_2$ may be a good indication of the quantity of dye removed from the effluent. C.O.C.

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Emulsions in Theory and Practice

C. G. Sumner Chem. and Ind., (20 Aug 1960) 1066–1070

An outline is given of the interplay of empirical knowledge and scientific theory in the historical development of emulsions. Three main theoretical problems—the influence of the emulsifying film on (a) phase relationship, (b) stability, and (c) bulk properties of an emulsion—are discussed. W.K.R.

Builders and Fillers in Household Detergents

N. Pilpel Research, 13 (Sept 1960) 336–343

An account of the builders, fillers and other materials which, for technical and economic reasons, are incorporated in household synthetic detergents. 25 references. C.O.C.

Anti-static Agents in the Textile Industry

A. E. Henahall J.S.D.C., 76 (Sept 1960) 525–535

PATENTS

Detergent

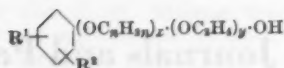
U.S. Sec. of Agriculture USP 2,915,473 (17 Apr 1956)

Mixtures of long-chain alkyl sulphates and α -sulphonated fatty acids have synergistic detergent properties. C.O.C.

Alkylphenoxypolyoxyglycol Non-Ionic Surfactants

Dow Chemical Co. USP 2,915,559 (10 Dec 1956)

Compounds of formula—



(R^1 and $R^2 = H$ or Alk and together contain 6–10 C; $n = 3$ or 4; $x = 3-5$; $y = 4-6$; $nx > 3y$) are readily obtained by treating an alkylated phenol with propylene in butylene oxide (3–5 mol. oxide per mol. phenol) and then treating the product with ethylene oxide (4–6 mol. per mol. original phenol). They are very powerful wetting agents. C.O.C.

N-(Polyoxa-alkyl)-perfluoroalkanesulphonamides—Non-ionic Surfactants

Minnesota Mining & Manufacturing Co.

USP 2,915,554 (23 July 1957)

Compounds of formula $R^1SO_2NR^2R^3$ ($R^1 =$ perfluoroalkyl of 4–12 C; $R^2 = H$, Alk or R^3 ; $R^3 = -(CH_2)_m$, $(OCH_2CHR^4)_n$, OR⁴ ($m = 2-3$; $n = 2-20$; $R^4 = H$ or CH_3), e.g. $C_8F_{17}SO_2(CH_3)N-C_2H_4-(OCH_2OCH_2)_5-OH$, show high surface activity and are particularly useful for emulsifying fluorochemical monomers. C.O.C.

Colloidal Dispersions of Silica in Oil for Treating Textiles

Monsanto

BP 840,372 (16 Feb 1956)

When silica is dispersed in oils to form fluid colloidal dispersions it retains its ability to improve interfibre friction.

BP 842,027 (26 June 1957)

Aqueous emulsions of the above dispersion are particularly suitable as lubricants in the production of yarns from wool and wool blends. C.O.C.

Softening Agents

Chemische Werke Witten

BP 842,842 (Germany 20 Jan 1958)

Mixtures of water-insoluble fatty acid alkanolamides with compounds of formula—



(R^1 , R^2 and $R^3 =$ same or different Alk; $R^4 =$ Alk, aralkyl or hydroxyalkyl; $X =$ anion; $n = 1-4$) applied to textiles impart a soft, full handle which is water-resistant. The treated textiles have good fastness to light and the colours of direct dyes are only slightly altered. C.O.C.

Textile Size

FBY

BP 843,185 (Germany 18 Nov 1955)

A non-aqueous solution of a benzene-soluble copolymer of an acid ester of an α , β -ethylenically unsaturated aliphatic dicarboxylic acid with an alcohol of > 8 C, e.g. octadecylmaleate, a neutral ester of a copolymerisable vinyl compound containing Alk of > 8 C, e.g. vinyl stearate and another copolymerisable vinyl compound, e.g. styrene, is used. It does not resinify after application even when the sized yarn is kept for long periods. C.O.C.

Vinyloxyalkylamidonitriles

Rohm & Haas Co.

BP 840,170 (U.S.A. 29 Nov 1955)

Compounds of formula $CH_2CHOZN(CR^1O)ACN$ ($Z =$ alkylene of 2–6 C; $A =$ cyclohexylidene, CH_3 , $CH(CH_3)$, CH_2CH_2 , $CH(CH_3)CH_2$, $CH_2CH(CH_3)$, $CH(CH_2CH_3)_2$, CHR^2 ($R^2 =$ Alk or 1–8 C), or $C(R^2)(R^3)$ ($R^2 = CH_3$ or C_2H_5); $R^1 = H$, Alk of 1–18 C, subst. or unsubst. Ar of 6–10 C), e.g. α -(N- β -vinyloxyethylacetamido)propionitrile, are useful as fungicides, insecticides, lubricating and softening agents for leather and textiles, sizes, etc. C.O.C.

Vinyl Sulphides of Substituted Ureas and Thioureas and Polymers and Copolymers

Rohm & Haas Co.

BP 842,001 (U.S.A. 15 July 1955)

Describes four types of novel monomeric ureido- and thioureido-vinyl sulphides and their polymers and copolymers. Both the monomers and polymers have very wide fields of use in the textile, paper, paint and lacquer, etc., industries. Thus N-ethyl-N'-methyl-N'-2-(vinylthio)ethyl urea (obtained by treating 2-(N-methylamino)ethyl vinyl sulphide with ethylisocyanate) copolymerised with 9 times its weight of acrylonitrile yields a product which has much better affinity for dyes than polyacrylonitrile and also has much better resistance to weathering. C.O.C.

Anti-static Composition

Mitsubishi Rayon Co.

USP 2,917,401 (20 Dec 1955)

The colloidal solution obtained by treating a mixed solution of an alkyl acetate and a monovalent aliphatic alcohol with a silicon halide in presence of water is a good anti-static agent for use on hydrophobic materials, e.g. synthetic polymer fibres, glass fibres, acrylic or vinyl resins, etc. C.O.C.

Bleaching Compositions

Unilever

BP 839,715 (8 Nov 1957)

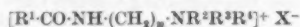
Bleaching agents containing inorganic per salts and arylcarboxylic esters can cause fading of dyed materials repeatedly bleached by them. The tendency to cause fading is to some degree related to the titre of Na thio-sulphate given by the ester in the test described in BP 836,988 (J.S.D.C., 76 (Sept 1960) 569). This tendency to cause fading may be reduced by using 4–20 atoms of available oxygen for each molecule of ester and by using esters of titre < 4 ml. C.O.C.

Combined Rot-proofing and Softening Agents

Chemische Werke Witten

BP 842,829 (Germany 20 Jan 1958)

Compounds of formula—



($R^1 =$ Alk of 7–21 C; R^2 and $R^3 =$ Alk of 1–3 C; $R^4 =$ Alk, aralkyl or hydroxyalkyl; $n = 2, 3$ or 4; $X =$ anion) treated with Zn pentachlorophenolate in a molecular ratio of $< 2.5:1$, respectively, impregnated into textiles impart both a rot-proof finish and a soft handle. Thus lauric acid is treated with dimethylaminopropylamine at 180°C. and then with water, benzyl chloride and isopropyl alcohol until a water-soluble product is obtained. This is then treated with aqueous Zn pentachlorophenolate for several hours at 80°C. The highly viscous, homogeneous paste obtained readily dissolves in water. C.O.C.

Reducing Agents for Vat and Sulphur Dyes

BASF

BP 837,940 (Germany 29 July 1958)

Compounds of formula $A_z \cdot N \cdot D_y$ ($A = H$ or Alk; $D = -CEG-SO_2-Z^+$ ($E =$ Alk; $G = H$ or Alk; $Z =$ alkali metal or NH_4^+); $z = 0, 1$ or 2; $y = 1-3$; $x + y = 3$), e.g. $[(O_2S(CH_2)_3CH_2)_3N \cdot CH_2CH_2N(CH_2CH_2SO_3)_3]_4 \cdot K^+$ are reducing agents for vat and sulphur dyes. At ordinary temperatures, even in presence of strong alkali, they do not reduce vat dyes but do so rapidly at $> 50^\circ C$. They enable dyes which are not reduced by cold sodium dithionite to be applied in a quick heat-fixing process, e.g. the dye is padded on to the cloth, treated with the above compound, immediately treated for 30 sec. in air-free steam at 108°C. without excess pressure, and then re-oxidised. C.O.C.

Resin Finish for Cellulosic Fabric

Quaker Chemical Products Corp.

USP 2,917,411 (12 June 1957)

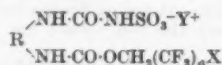
The condensates of 2-hydroxyethylamine, urea, and formaldehyde impart crease recovery and dimensional stability to cellulosic fabrics without imparting retention of chlorine when they are blended. C.O.C.

Fluorine Compounds for Rendering Textiles Oil-repellent

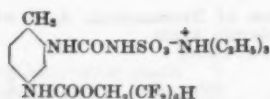
DuP

USP 2,917,409 (31 Dec 1958)

Compounds of formula—



($R =$ bivalent organic radical inert to isocyanate groups; $X = H$ or Hal; $Y =$ tertiary aliphatic amine ion; $n = 4-12$) when padded into textiles and then dried and cured impart an oil-repellent finish. Thus 2,4-toluene diisocyanate (17.4 parts) and 1,1,9-trihydroperfluorononyl alcohol ($H(CF_2)_9CH_2OH$) (43.2) were dissolved in anhydrous dimethylformamide (57) and pyridine (1.0) was added. The temperature immediately rose by 8°C. In a separate vessel sulfamic acid (12.6), triethylamine (14.4), and dimethylformamide (29) were mixed and added to the first solution. The mixture was heated to 40°C. for one hour to yield a solution of—

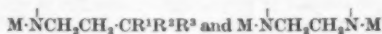


This solution was brought to < 3% strength by dilution with water. Cotton and nylon cloths impregnated with their own weight of the diluted solution, air-dried and baked at 107°C. for 90 sec. received an oil-repellent finish which resisted washing with detergent in an automatic washer. C.O.C.

Reaction Products of 1-Aziridinyl Compounds with Compounds containing Active Methylene Groups—Flame-resisting Finishes for Textiles
U.S. Secretary of Agriculture

USP 2,917,492 (4 Dec 1956)

Compounds containing more than one 1-aziridinyl group attached directly to a non-metallic atom react with compounds that contain more than one active H atom attached to C atoms which are in α -position to an electronegative group to yield polymers containing the recurring connecting structures—



(M = P or S, R¹ = H, Alk or Ar; R² = electronegative group; R³ = electronegative group Alk or Ar). These polymers may be either liquid or solid. They are flame-resistant and when deposited in the interstices of hydrophilic fibrous materials reduce their flammability. C.O.C.

Water-dispersible Polyester-Aminoplast Condensates—Coatings, Binders, etc.

Rohm & Haas Co. USP 2,915,486 (5 Mar 1956)

The condensates of (1) a polyester of mol.wt. 800–1500 containing hydroxyl groups which can react with the second component and carboxylic groups which can be neutralised with ammonia, amines or quaternary ammonium compounds and (2) an organic solvent aminoplast, are particularly useful for coating textiles and as binders for pigment pastes for the printing or dyeing of textiles.

USP 2,915,487 (3 Mar 1958)

Condensates of (1) an alkyd of mol.wt. 2000–10,000 and containing available hydroxyl groups, (2) an organic-solvent-soluble, water-insoluble aminoplast, and (3) a polyester of the type described above, have similar properties in that they have excellent curing properties, yield films of good cohesion, flexibility and toughness, and yet are self-dispersible in aqueous systems. C.O.C.

Phosphorus-containing Polymers from 1-Aziridinyl Compounds for imparting Flame-resistance to Organic Materials and Reducing Felting Power of Wool

Albright & Wilson BP 837,709 (U.S.A. 5 June 1956)

The polymers obtained by heating aziridinyl phosphine oxides or sulphides with methylol phosphorus compounds are useful for imparting flame-resistance to organic materials. Treatment of wool with the monomers or their precondensates with methylol compounds reduces its felting power.

BP 837,710 (U.S.A. 4 Dec 1956)

The polymers obtained by treating compounds containing more than two 1-aziridinyl groups attached to pentavalent P atoms with a monohydric phenol, carboxylic acid and/or a nitrile or sulphone compound containing at least two active H atoms attached to C atoms, impart flame-resistance to organic materials. C.O.C.

Hair-waving Composition

Gillette Co. BP 839,923 (U.S.A. 27 Mar 1957)

$\alpha\alpha'$ -Dimercaptoallipic acid and its salts, esters, and amides are effective waving agents for the cold permanent-waving of hair. C.O.C.

Aqueous Binders for Use in making Bonded-fibre Fabrics

Rohm & Haas Co. BP 840,127 (U.S.A. 13 Aug 1956)

Predominantly non-proteinaceous fibres incapable of felting are bound together by use of an aqueous dispersion of (a) a water-insoluble linear polymer of monoethylenically unsaturated monomeric units comprising < 3% by weight of a compound of formula $\text{CH}_2=\text{CR}^1\text{O}-\text{CAR}^2\text{OH}$

[R¹ = H or CH₃; R² = straight or branched chain alkylene of 2–6 C or (C_nH_{2n})_mC_nH_{2n} (n = 2 or 3; m = 1, 2 or 3; A = O or NH)] and (b) an aminoplast. The linear polymer must have been cross-linked to make it insoluble and infusible. It does not affect the colour of the fibres, and does not tend to migrate or give a rubbery or papery handle. C.O.C.

Vegetable Tannins. IV—Divi Divi Pods (C.I. Natural Brown 6) (IV p. 639)

Rendering Dyes and similar Powders Non-dusting (IV p. 643)

Aqueous Dispersions for Coating Fibrous Materials (IV p. 643)

Synthetic Polymers of Petroleum Origin for Coatings (X p. 650)

Silver Thiocyanate as a Bactericide (X p. 651)

Bactericidal Synthetic and Proteinaceous Fibres by Use of Neomycin (X p. 651)

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Relationship between Molecular Weight of Dye and Entropy of Dyeing

K. Odajima

J.S.D.C., 76 (Sept 1960) 550–551

Why are Substantive Dyes Substantive? II—Behaviour of Dyes in Electrolyte-free Solutions

A. Schaeffer *Melliand Textilber.*, 41 (Aug 1960) 988–992

Only dyes that by reason of their chemical constitution are able to form isoelectric ions can aggregate in electrolyte-free solutions and only such dyes will dye cellulose to a measurable degree in electrolyte-free solutions. W.M.

Temperature Dependence of Photoisomerisation Equilibria. I—Azobenzene and the Azonaphthalenes

E. Fischer

J. Amer. Chem. Soc., 82 (5 July 1960) 3249–3252

Solutions of azobenzene (I), 1,1'-azonaphthalene (II), and 2,2'-azonaphthalene (III) in organic solvents (including propen-1-ol, methylcyclohexane, methylcyclopentane and isononane) have been irradiated with light at specific wavelengths and a photoequilibrium between the respective *cis* and *trans* isomers established. The position of the photoequilibrium was found to be temperature dependent below 0°C. for (I) and (III) and below –20°C. for (II). Below these temperatures, thermal isomerisation is absent. Cooling shifts this equilibrium towards the *trans* isomer, the extent of the shift depending on the nature of compound, solvent and wavelength of irradiating light, e.g. irradiation of (III) in methylcyclohexane at 365 m μ . results in 20% *trans* form at –20°C. and 32% *trans* form at –125°C. Quantum yields of photoisomerisation indicate a strong dependence on temperature. F.J.

“Thermal” Rearrangement of Hydrazo Compounds.

III—Kinetics and Mechanism of Rearrangement of 2,2'-Hydrazonaphthalene in Polar Solvents

H. J. Shine and J. C. Trisler

J. Amer. Chem. Soc., 82 (5 Aug 1960) 4054–4058

The effect of different solvents (ethanol, aq. ethanol, acetone and tetrahydrofuran) has been studied on rearrangement of 2,2'-hydrazonaphthalene (I), which undergoes a type of benzidine rearrangement usually attributed to acid catalysis, free radicals or a thermal effect. Yields of 80–85% of 2,2'-diamino-1,1'-binaphthyl and 15–20% 3,4,5,6-dibenzocarbazole were obtained, independent of solvent used. Rates of rearrangement of (I) were measured at several temperatures in these and other solvents. Rates in anhyd. alcohol were faster than in acetone, dioxan, tetrahydrofuran and pyridine, and rates in aq. alcohol increased with concn. of water. Activation energies and entropies were found to be 23.2, 20.5 and 30.9 kcal. mole⁻¹ and –13.4, –4.6 and –1.6 cal. mole⁻¹ deg.⁻¹ in ethanol, dioxan, and pyridine, respectively. The rearrangement in hydroxylic solvents may involve a polar transition state, formation of which is enhanced as solvent becomes more polar, i.e. aqueous, whereas in non-polar solvents transition state is independent of solvent. A reaction scheme is given: F.J.

Effect of Steric Factors on Properties of Dyes Containing the Diphenyl Nucleus. XIV—Colour and Affinity for Cotton of some Azo Dyes—Derivatives of Diphenyl, Dibenzyl, *Trans*-Stilbene, Diphenylacetylene, and Azobenzene

B. M. Krasovitskii, D. G. Pereyaslova, and N. I. Titarenko
Ukrain. khim. zhur., 26, No. 1 (1960) 73-77

The effect of conjugation on colour and affinity for cotton of aq. soln. of azo dyes, obtained from 4,4'-diamino deriv. of dibenzyl, *trans*-stilbene, diphenylacetylene, and azobenzene has been investigated spectrophotometrically and the results compared with earlier findings (B. M. Krasovitskii *et al.*, *Ukrain. khim. zhur.*, 20 (1954) 646) for corresponding bisazo dyes—deriv. of diphenyl and *p*-terphenyl. Deriv. of 4,4'-diaminoazobenzene differ from their 3,3'-analogues, the latter showing considerable hypochromic displacement of absorption max. and decrease in affinity for cotton.

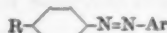
G.J.K.

Synthesis and Properties of Acid Monoazo Dyes (7th Communication)

L. N. Nikolenko, O. I. Eremina, E. N. Karpova, I. F. Mikhailova, and L. S. Kobrina

Zhur. priklad. khim., 33 (July 1960) 1617-1623

Fastness properties of simple acid monoazo dyes—



prepared from alkyl-(4-chlorophenyl)ketones, are related to the nature of R (R = H; C₂H₅ to C₁₂H₂₅; or sec. C₄H₉; C₈H₁₇; C₁₁H₂₃; Ar = 1-(*p*-phenylsulphonate)-3-methyl-5-pyrazolone; or acetyl of H-acid). Using wool as substrate, it is found that increasing the chain length of R affects favourably fastness to wet treatment but decreases fastness to rubbing, light, and weather. Increase in chain length of R up to C₁₀-C₁₁ increases dye uptake but lowers dye levelling properties. Fastness properties of dyes having large R depend upon size rather than structure of R. Detailed results of fastness testing and brief preparative notes are given.

G.J.K.

Polarography of Azo Dyes having a Pyrazolone Component

M. Kožený

Šorník věd prací, Vysoká škola chem.-technol. Pardubice (1959) 167-172

The polarography of 7 azo dyes made with 1-phenyl-3-methyl-5-pyrazolone as the coupling component showed considerable shift of the half-wave potentials toward negative values when compared with similar, purely aromatic dyes.

C.O.C.

Absorption Spectra of Dyes. VI—Steric Effects in Conjugated Systems of Benzidine Disazo Dyes and their Copper Derivatives

T. Kobayashi, Y. Tanizaki, and N. Ando

Bull. Chem. Soc. Japan, 33 (July 1960) 913-917

From absorption spectra of 5 benzidine disazo dyes and their copper deriv. in aq. soln. at room temp., it is shown that those dyes which have sulphonic groups adjacent to the azo groups exhibit strained conformations due to repulsion between the sulphonic groups and the unshared electrons of nitrogen. The absorption intensities of the copper deriv. are nearly equal and are not affected by the positions of the sulphonic groups. Moreover, they are near to those of the non-copper dyes which have the sulphonic groups adjacent to the azo groups. It appears, therefore, that the introduction of copper produces the same extent of strain in conjugated systems as do the sulphonic groups.

H.H.H.

Some Acetoacetates of Aromatic Amines and Azo Dyes derived from them

P. M. Heertjes, H. C. A. van Beek, and H. A. Schmidt

Melliand Textilber., 41 (Mar 1960) 320-322

The preparation of acetoacetic acid deriv. of aromatic amines starting from diketene is described. Several cpd. obtained in this way were used to produce azo dyes by the usual methods. Acid dyes, acid dyes for afterchroming, and direct dyes were prepared and their fastness properties determined.

W.M.

Arylamination of Bromaminic Acid with Amino-naphtholsulphonic Acids

J. Reichel and A. Balint

Studii și Cercetări, Științe Chimice

(Timișoara), VI (Jan-June 1959) 87-94

Soln. of the sodium salt of bromaminic acid (1-amino-4-bromoanthraquinone-2-sulphonic acid) react with J-, *p*-, and H-acids (in aq. NaHCO₃ at 60-70°C. for 8 hr. and with a small amount of CuSO₄) to form a series of blue dyes of beautiful hue and excellent affinity for wool. In consequence of an active coupling position in the amino-naphthol part of the mol., the above dyes can react with diazonium salts to afford another set of violet dyes. The cpd. involving J-acid and phenyl-*p*-phenylenediamine intermediates (as coupling components) have good affinity for cotton also. Coupling on the fibre gives the same result as dyeing with the coupled dye.

H.H.H.

Synthesis, Purification, and Behaviour of the Dye Sodium 1,8-Aminonaphthol-2-(4'-nitrophenylazo)-3,6-disulphonate

A. Demian, I. Ipsilante, and W. Schmidt

Studii și Cercetări, Științe Chimice

(Timișoara), V (July-Dec 1958) 115-121

A simple method of preparing the title dye is reported. It is obtained in high concn. and free from coloured by-products and inorg. salts; the concn. may be determined by pptn. of its benzidine salt. The dye is useful as an acid-base indicator with range pH 5.6-7.2 from rose to violet and pK 6.2; λ_{max} = 5200 Å. (in acid medium) and 5650 Å. (at pH 8).

H.H.H.

Separation Components in the Synthesis of Azo Dyes V—Absorption Spectra of Some Intermediate Azo Dyes

I. Reichel and A. Demian

Studii și Cercetări, Științe Chimice

(Timișoara), V (Jan-June 1958) 67-75

Previous work (cf. J.S.D.C., 74 (Aug 1958) 598) has been concerned with the preparation of a series of asymmetrical azo dyes in which conjugation throughout the entire mol. has been interrupted by separation components. This work is now continued by a determination of the absorption spectra of some dyes of simple structure which contain the end yellow and blue components of the former asymmetrical dyes, and then that of a dye in which these end components are united without the intervention of a central separation component. For this purpose, the yellow component is represented by benzene-azosalicylic acid and the blue by a dye in which H-acid has been first coupled with diazotised *p*-nitroaniline and then with diazotised aniline. The united dye is formed by using diazotised aminosalicylic acid in place of diazotised aniline.

VIII—Behaviour of 1,5-Disazoanthraquinone as a Central Component

I. Reichel and W. Schmidt

Ibid., V (July-Dec 1958) 87-101

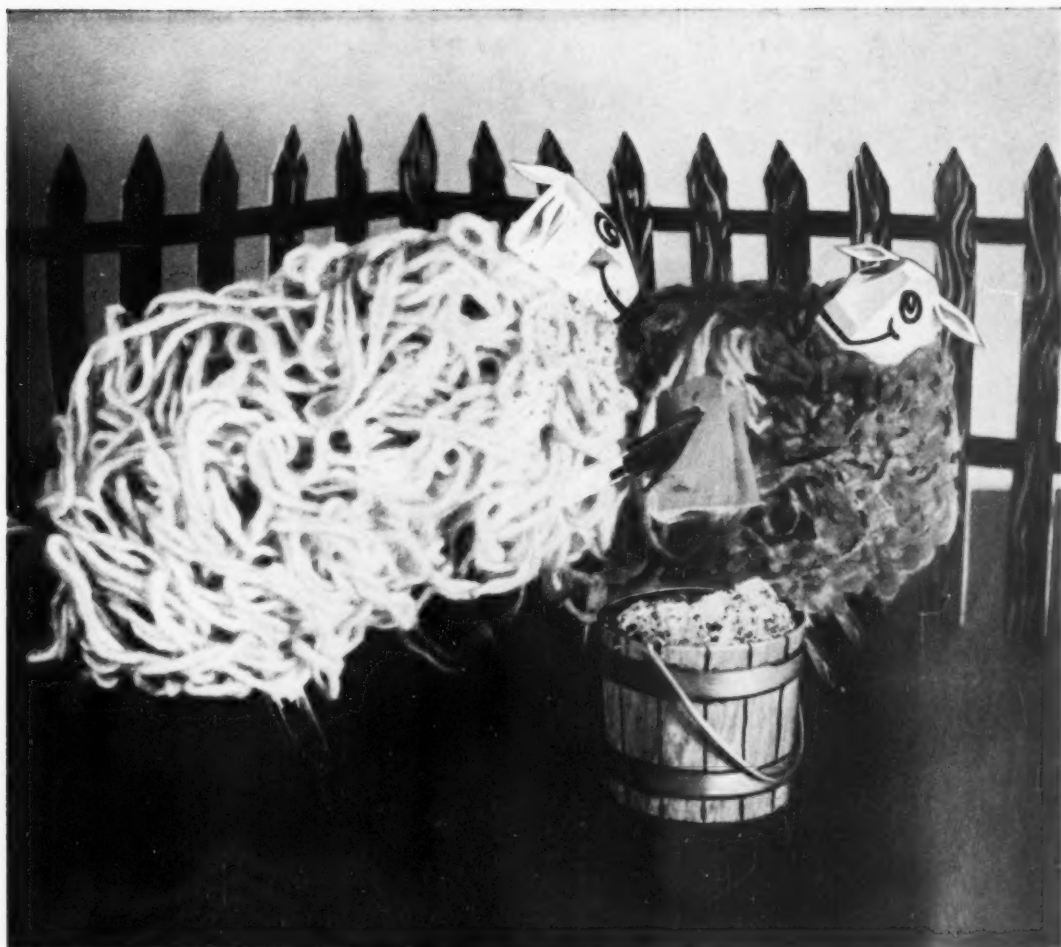
Diazotised 5-oxalylamino-1-aminoanthraquinone was coupled with the yellow end-components phenol and 1-phenyl-3-methyl-5-pyrazolone but not with salicylic acid; the azo dyes thus formed, when hydrolysed and diazotised, were coupled with the blue end-component 1,8-amino-naphthol-2(4'-nitrophenylazo)-3,6-disulphonic acid. Bis-diazotised 1,5-diaminoanthraquinone was mono-coupled with salicylic acid and then with the blue component above. All three asymmetric triazo dyes were deep green and were only sparingly sol. in water. Attempts to dye them in a vat using sodium dithionite (hydros) resulted in splitting of the azo groups. The reddish colour of the central anthraquinone component appeared to influence appreciably the resultant colour of these asymmetric dyes.

IX—Absorption Spectra of Some Azo Dyes having Diazotised 1,4- and 1,3-Phenylene Diamine as Central Components

I. Reichel and A. Demian

Ibid., 103-114

Absorption spectra are reported for the following six azo dyes—Salicylic acid \leftarrow tetrazotised *p*-(and *m*)-phenylenediamine \rightarrow (2) H-acid (1) \leftarrow diazotised *p*-nitroaniline, *p*-(and *m*)-aminobenzene-azo-salicylic acid, and diazotised *p*-(and *m*)-aminobenzene \rightarrow (2) H-acid (1) \leftarrow diazotised *p*-nitroaniline. The triazo system which contains the *p*-phenylenediazazo component admits of conjugation throughout the entire mol. with absorption



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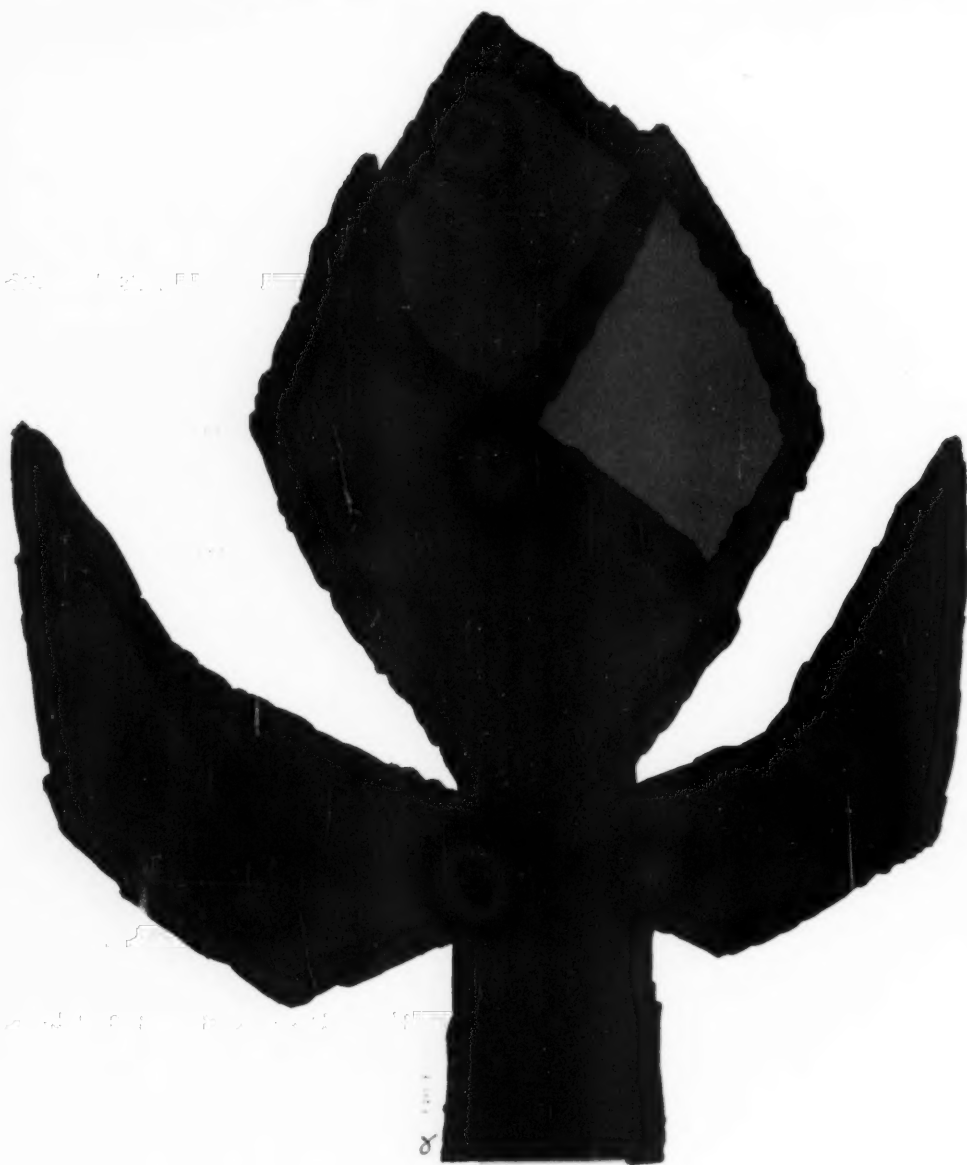
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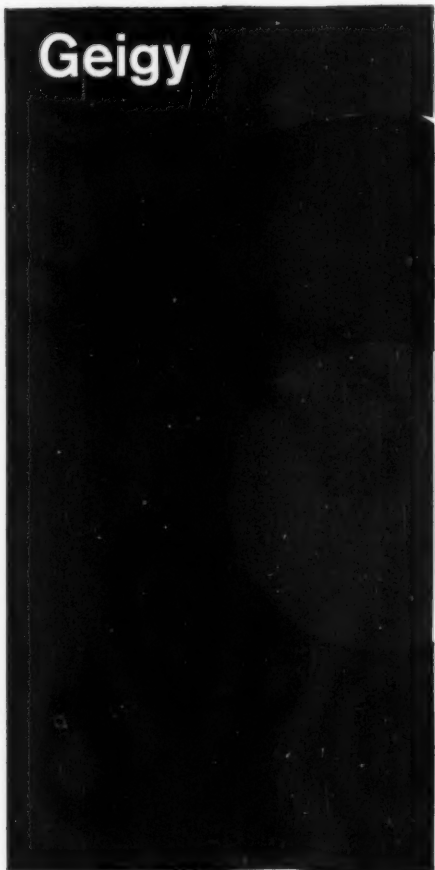
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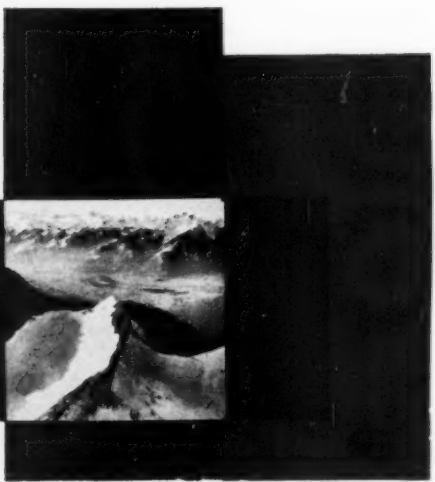
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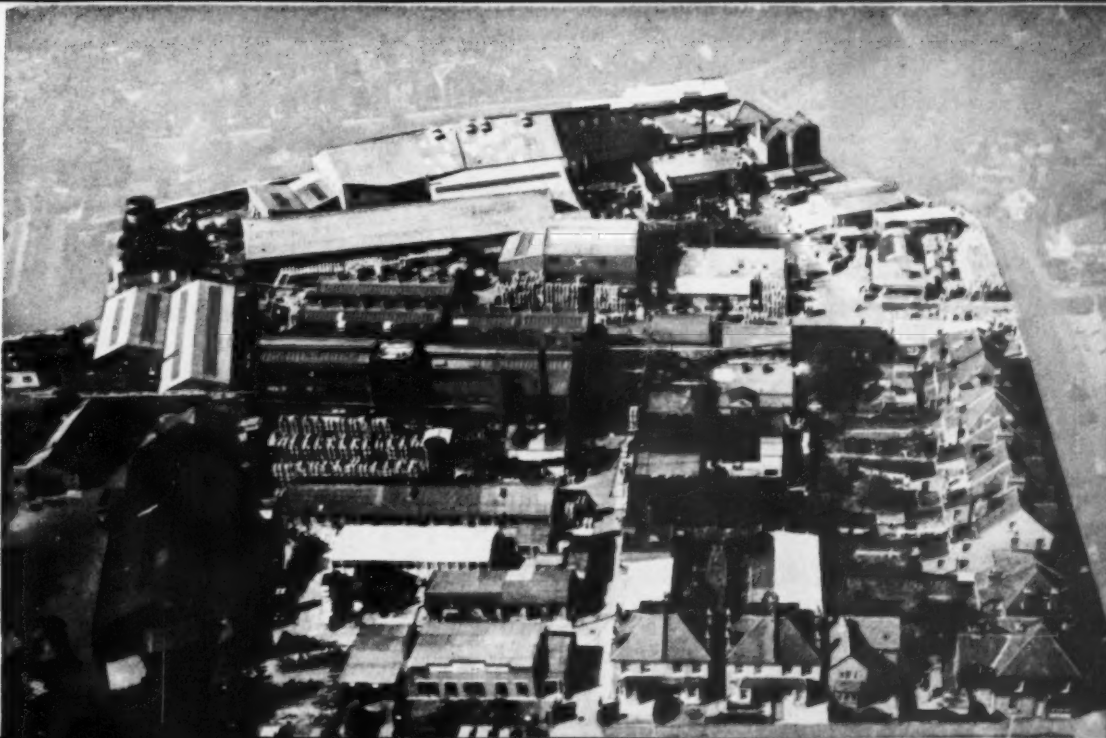
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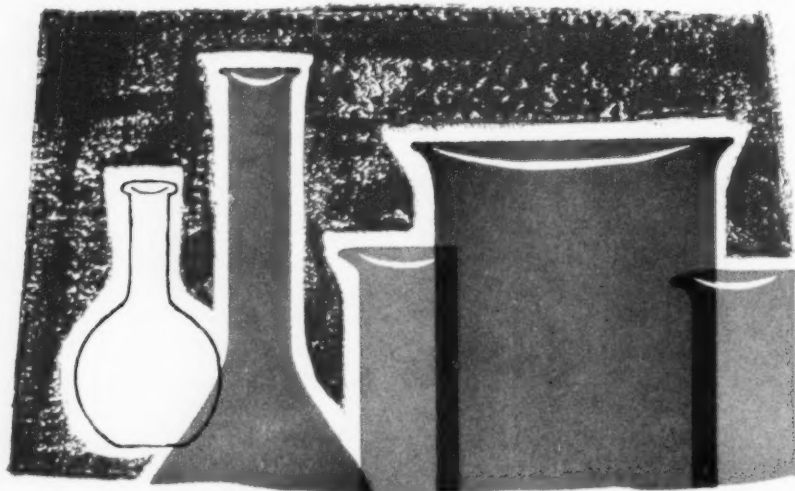
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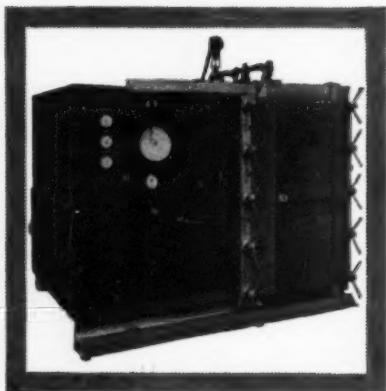
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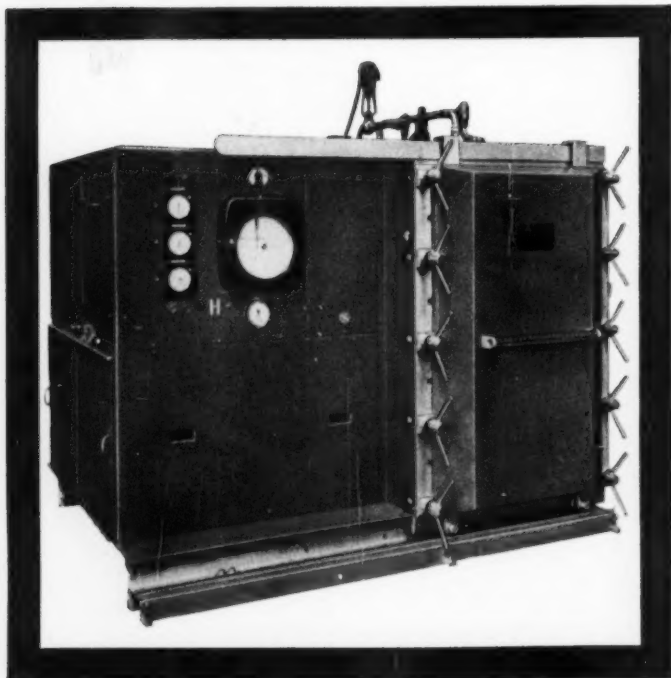
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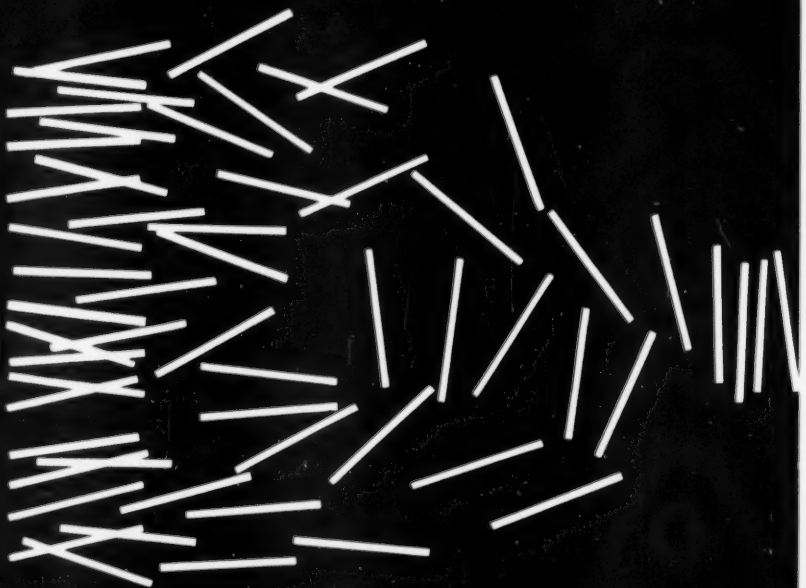
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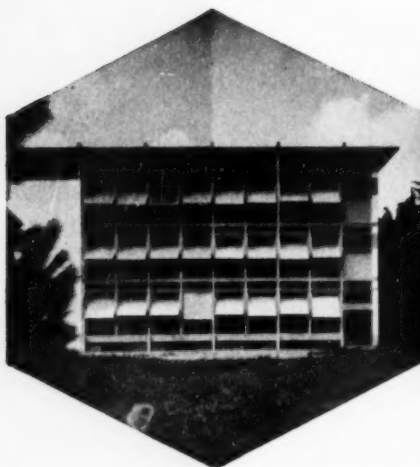
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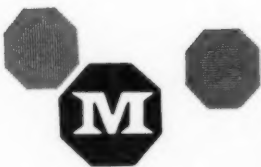
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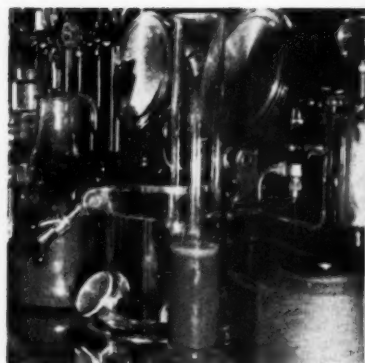
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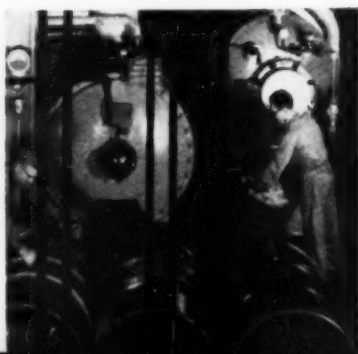
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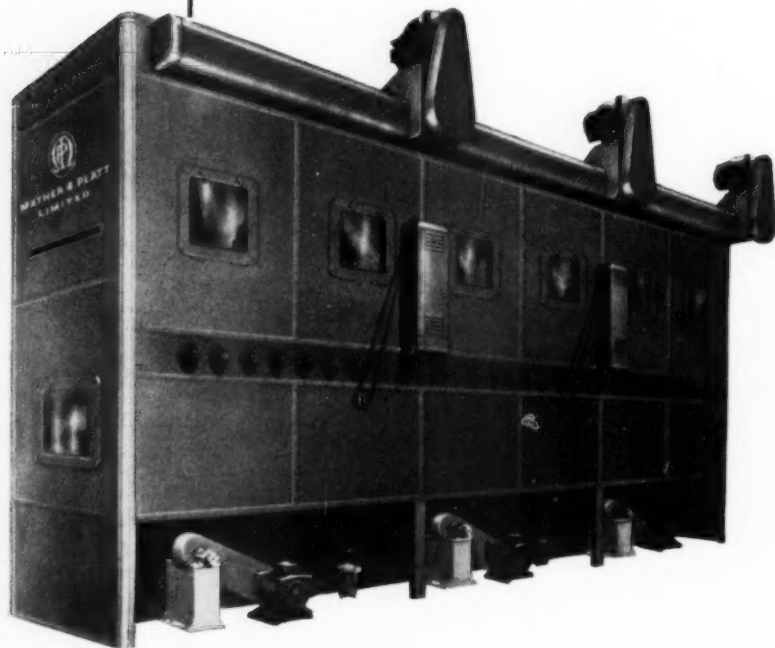
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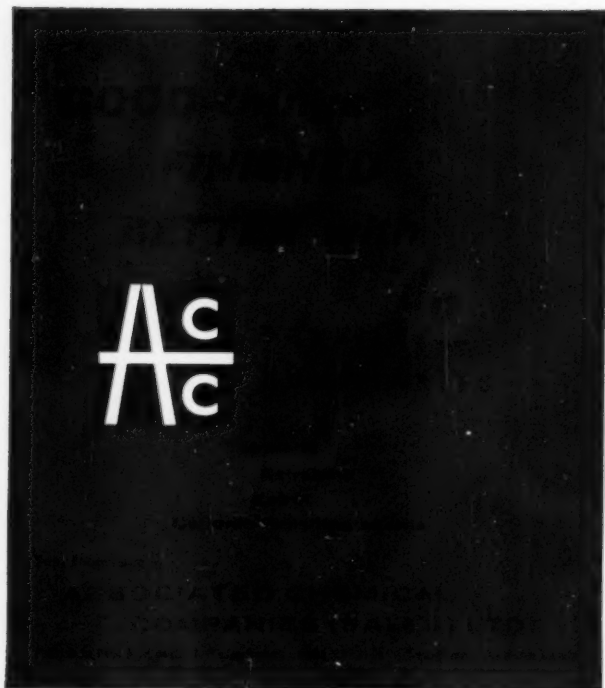
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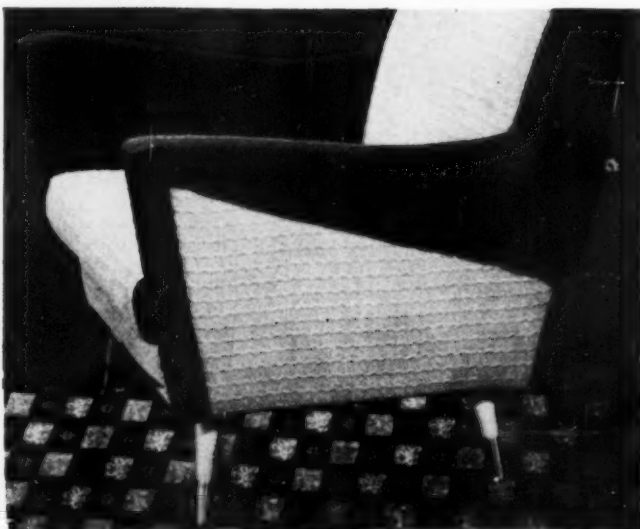
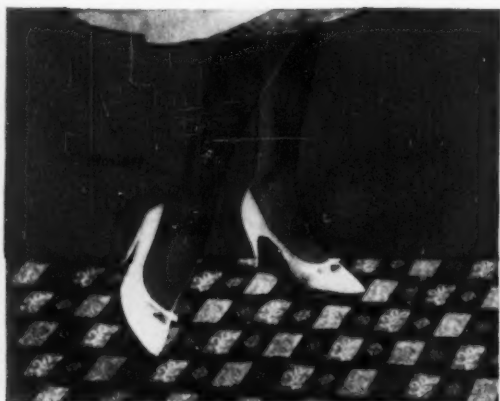
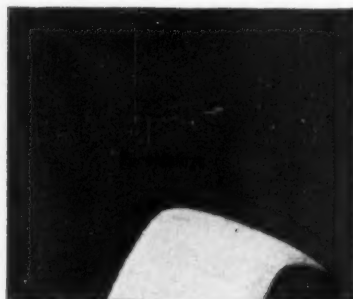
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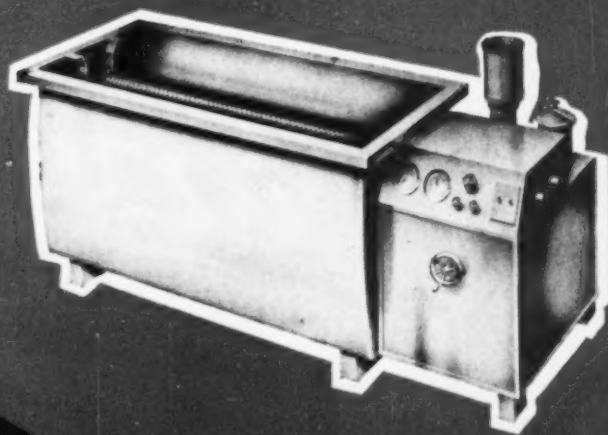


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displaced in a bathochromic direction. On the other hand, the system with a central *m*-phenylenedisazo component has its conjugation interrupted with the resultant colour being an intramol. addition of the colours of the end-components, although with some intensification of the absorption.

XII—Absorption Spectra of Some Anthraquinone-1,5-Disazo Compounds

J. Reichel and W. Schmidt

Ibid., VI (Jan–June 1959) 77–85

New absorption spectra are reported which establish that the anthraquinone-1,5-disazo group is a central separation component. H.H.H.

Dyeing Properties of a Triazole Derivative of Congo Red

K. Nishida

J.S.D.C., 76 (Sept 1960) 551–552

Formation of a Helical Complex by a Sulphonphthalein Dye

D. M. Blow and A. Rich

J. Amer. Chem. Soc., 82 (20 July 1960) 3572–3574

The changes in structure of Bromphenol Blue as a function of pH have been examined and wavelengths of max. absorption of each structure have been found. It is suggested that at pH 10–14 the colourless soln. contains the monovalent ion of the dye in which a hydroxyl group remains attached to the central carbon atom. On acidification to pH 4 a white fibrous ppt. is formed, confirming original observation of V. K. La Mer and E. S. Amis, *Science*, 90 (1939) 90. An X-ray diffraction pattern of this ppt. showed that the complex contains a helical arrangement of molecules. This behaviour is specific to this dye, since attempts to produce the same effect with tetraiodosulphonphthalein, Bromcresol Green, Bromcresol Purple, Bromthymol Blue, Phenol Red and Eosine (C.I. Acid Red 87) were unsuccessful. The dependence of structure of Bromphenol Blue on pH is shown diagrammatically. F.J.

Phosphorescence of Adsorbed Acriflavine (C.I. 46000)

J. L. Rosenberg and D. J. Shombert

J. Amer. Chem. Soc., 82 (5 July 1960) 3252–3257

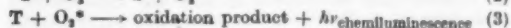
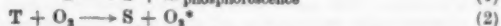
The phosphorescence of purified acriflavine adsorbed on silica gel has been studied below room temperature by flash technique, observing the luminescence by means of a photomultiplier connected to an oscilloscope, the sweep trace being photographed. Phosphorescence decay of acriflavine was non-exponential and strictly first order over a wide range of conditions. Interference due to phosphorescence originating from silica gel was minimised by removal of all radiation < 440 mμ. from the activating light. The effect of dye concn. on decay rate and dependence of decay rate on the excitation wavelength and the luminescence observation have been studied. Activation energy for α -phosphorescence indicated that greater nuclear displacement occurred in the triplet than in the fluorescent singlet state. Non-radiative decay from triplet to ground states was less than three times as fast as the direct radiative transition. F.J.

Reaction of Oxygen with Electronically Excited Acriflavine (C.I. 46000)

J. L. Rosenberg and D. J. Shombert

J. Amer. Chem. Soc., 82 (5 July 1960) 3257–3261

Interaction of oxygen and photo-excited acriflavine adsorbed on silica gel has been studied by flash technique over a wide range of conditions. Reactions with oxygen were carried out by addition of the gas to sample before or immediately after flash excitation or with continuous irradiation in presence of oxygen. Proposed mechanisms are given below where S and T are the ground and first triplet state of acriflavine, respectively, and O_2^* is vibrationally excited O_2 —



From -160° to $+10^\circ$ C. reaction (2) is collision-limited and almost independent of temperature. Below -160° C. rate-controlling step of (2) is the desorption of O_2^* with an activation energy of 5–6 kcal., and in this range (2) competes poorly with (1). Practically all O_2^* is

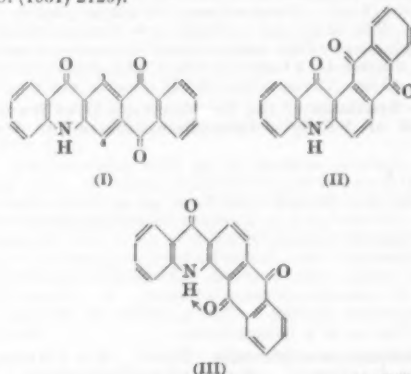
deactivated by the surface (4) or by ground state of acriflavine (5). Reaction (3) uses < 10% of O_2^* and is accompanied by a good yield of chemiluminescence from oxidation product. F.J.

N-Methyl Derivatives of Phthaloylacridones

W. Bradley and H. Kaiwar

J.C.S., (July 1960) 2859–2864

Twelve new deriv. of 3,4-phthaloylacridone (III) are described, and the structural influence of *N*-methylation on their m.p., solubility, and light absorption examined. Also included are the effects of *N*-methylation on the properties of 1,2-(II)- and 2,3-phthaloyl-(I) acridone and of 5,6-phthaloyl-1,2-benzacridone. *N*-methylation increases the solubility in org. solvents and lowers m.p. throughout the series; in the case of I, the occurrence of intermol. H-bonding is prevented between the $-NH-$ and $-C=O$ of two or more mol., the acidity of II is destroyed, and the intramol. H-bonding in III is greatly restricted by interference with mol. planarity. Further, *N*-methylation notably reduces the affinity for cotton of the parent cpd., and indicates that the vat-dyeing properties of the latter are due to H-bonding between NH -groups and cellulose oxygen. The above relationships are parallel to those previously observed with indanthrone (cf. J.C.S. (1951) 2129).



H.H.H.

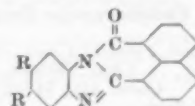
Dyes Derived from Imidazole. II—Monoaroylene Imidazole Dyes Derived from Teramines of the Benzene Series

J. Arient and J. Marhan

Collection Czechoslov. Chem. Commun., 25 (1960) 124–129

Chem. Abs., 54 (25 May 1960) 9894

The dye from coupling diazotised sulphanilic acid to *N,N*-bis-(*p*-toluene sulphonyl)-*m*-phenylenediamine gave, with Na hydrosulphite, 1,5-bis-(*p*-toluene sulphonyl)-2,4-diaminobenzene, and thence, by acid hydrolysis, sym-tetramino benzene (II) and the tetra hydrochloride (III). III refluxed in acetic acid with naphthalene-1,8-dicarboxylic acid (I) gave a crude product, chromatographed on Al_2O_3 and diluted with nitrobenzene, ethyl acetate and 1:1 benzene-cyclohexanol to give—



VI (R = H, R' = OAc), V (R = H, R' = NHAc) and predominantly IV (R = R' = NHAc). Addition of Fe/HCl to a mixture of 1,5-diamino-4,6-dinitrobenzene (VII) and (I), on refluxing in HOAc, gave IV, and similarly, 2,4-dinitroaniline and I gave, on Al_2O_3 (toluene), an unidentified product, VI, and (CHCl₃) V. The unsubst. compound (R = R' = H) (VIII) was one of the byproducts of the condensation of I with VII, followed by reduction of the NO_2 groups. Chromatography on Whatman No. 1 paper gave R_f values and fluorescence colours as follows— I, 0.75, violet; IV, 0.25, yellow; V, 0.45, yellow orange; VI, 0.6, green; VIII, 0.85, yellow green. The 7-methyl derivative of IV was similarly prepared from 3,5-bis-phenylazo-2,6-diaminotoluene. (See also FP 1,090,115, Chem. Abs., 53 (1959) 8645.) A.T.P.

Synthesis of Dyes with the Neocyanine Structure (Brooker-type), and Related Dyes

S. Kimura

III—Condensation of 3-Ethyl-2-methylbenzothiazolium Iodide and Nitrosobenzene, and the Dyes obtained from its Condensates

Bull. Chem. Soc. Japan, 33 (July 1960) 872-875

The title reaction, when performed in ethanol with piperidine as condensation agent, gave the phenyl nitrone of 3-ethyl-2-formylbenzothiazolium iodide instead of the expected anil. By refluxing an equimol. mixture of this cpd. and 3-ethyl-2-methylbenzothiazolium iodide in pyridine for 30 min., a mixture of 4 kinds of dyes was obtained and separated. These were bluish violet (IV), green (V), copper coloured (VI), and dark brown (VII) respectively. IV was identified as the known 3,3'-ethylthiacarbocyanine iodide dihydrate.

IV—Structure of the Dyes obtained by the Condensation of 3-Ethyl-2-methylbenzothiazolium Iodide with Nitrore or Anil Derivatives

Ibid., 875-879

Syntheses are described which establish V (see above) as 3,3'-diethyl-9-(2-benzothiazolyl)-thiacarbocyanine iodide, and VI (the so-called 3,3'-diethyl-9-(3-ethyl-1-thia-3-azoniainden-2-yl)thiacarbocyanine diiodide) as 3,3'-diethyl-9-(3-ethyl-2-benzothiazolyl)thiacarbocyanine diiodide. The latter cpd. is found to be formed initially in the early stage of the condensation; the reaction mechanism is discussed. VI changes into V when heated at 130°C. for ca. 12 hr. The structure of VII remains undetermined.

V—Synthesis of the Brooker-type Dyes from the Anil of 3-Ethyl-2-formylbenzothiazolium Iodide

Ibid., 879-881

An equimol. mixture of the title substance and 3,3'-diethyl-9-methylthiacarbocyanine iodide (I), when heated in pyridine at 80-100°C. for 5 min. gives the Brooker-type dye, 3,3'-diethyl-9-[2-(3-ethyl-1-thia-3-azoniainden-2-yl)-vinyl]thiacarbocyanine diiodide in good yield, and similar condensation of 3-ethyl-2-formylbenzothiazolium iodide with I affords 3,3'-diethyl-9-[2-(3-ethyl-1-thia-3-azoniainden-2-yl)-vinyl]thiacarbocyanine diiodide. The synthesis of Brooker-type dyes from anils is recommended.

H.H.H.

Anthraquinonesulphonate Dyes. X—1-Amino-2-sulpho-4-aryloxy-, -4-arylthioanthraquinone, 1,5-diamino-2-sulpho-4,8-bis(arylamino)-, -4,8-bis(aryloxy)- and 4-bis(arylthio)-anthraquinones

J. Degani and A. Tundo

Boll. sci. fac. chim. ind. Bologna, 17 (1959) 55-59*Chem. Abs.*, 54 (10 July 1960) 13667

1-Amino-2-sulpho-4-bromoanthraquinone was condensed with phenol, thiophenol or one of their $-\text{CH}_3$, $-\text{OCH}_3$, $-\text{Cl}$ or $-\text{OC}_2\text{H}_5$ derivatives to yield wool dyes. Similarly, 1,5-diamino-2-sulpho-4,8-dibromoanthraquinone was condensed with phenols, thiophenols or aniline or its derivatives. The monoaryloxy derivatives are orange and yellowish red dyes, the monoarylthio derivatives are reddish violet and the bis(aryloxy) and bis(arylthio) derivatives are blues. The monoarylthio dyes are faster to washing and light than the monoaryloxy dyes.

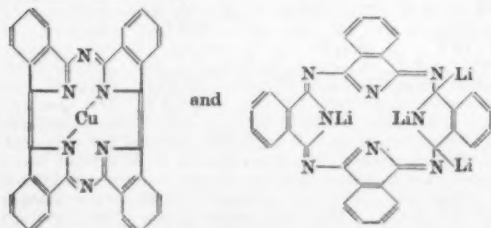
C.O.C.

Colour Value of Phthalocyanine Dyes and Pigments

V. F. Borodkin

*Izvest. Vysshikh Ucheb. Zavedenii, Khim. i**Khim. Tekhnol.*, 2 (1959) 424-429*Chem. Abs.*, 54 (10 July 1960) 13667

Synthesis of the dyes—



is described. Study of their properties and absorption spectra is described. Cleavage and shortening of the

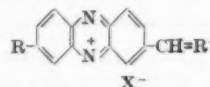
linkage in the tetra-azaporphin ring lead to shift of the absorption maximum toward shorter wavelengths, i.e. toward expansion of the colour range. C.O.C.

Cyanine Dyes from Phenazine Derivatives

A. I. Kiprianov and É. A. Ponomareva

Ukrain. khim. zhur., 26, No. 1 (1960) 78-85

Oxidative condensation of quaternary phenazinium salts with 2-methyl deriv. of benzthiazole; 6,7-benzobenzthiazole; benzazelenazole; 4-methylthiazole; benzoxazole; 4,5-benzobenzoxazole; 3,3'-dimethylindolenine; N-methylbenzimidazole, and quinoline gave 18 phenazino-cyanines of formula—



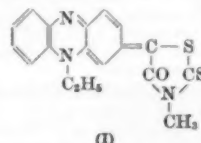
(R = H or CH_3O ; X = I, ClO_4 , SO_4). The absorption max. of these dyes in ethanol fall between 700-800 mμ. Preparation of corresponding asymmetrical dyes proved unsuccessful. Structural formulae, absorption max., yields, temp. of decomposition, and analytical data are tabulated. G.J.K.

Cyanine Dyes—Derivatives of Phenazine (2nd communication)

A. I. Kiprianov, É. A. Ponomareva, and Ya. P. Skavinskii

Ukrain. khim. zhur., 26, No. 2 (1960) 237-242

Oxidative condensation of quaternary salts of phenazine, phenanthrazine, and their methoxy deriv., with deriv. of rhodamine and pyrazolone gave seven merocyanines (M), e.g. I, in good yield—



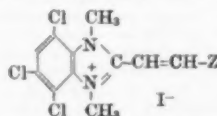
Absorption max. in chloroform occur at about 700 and 400 mμ. Action of dimethyl sulphate on M and subsequent condensation with quaternary salts of 2-methylbenzthiazole, 2-methylbenzoxazole, or quinaldine gave three trinuclear rhodacyanines, the absorption spectra of which lack clear definition. Condensation of methylmethoxy-sulphates of phenanthrazine, acenaphthylenequinoxaline and their respective 6- and 4-methoxy deriv. with quaternary salts of 2-methylbenzthiazole gave four monomethincyanines in good yield; the methoxy group caused considerable hypsochromic displacement of absorption max. G.J.K.

Cyanine Dyes from 2-Methyl-4,5,7-trichlorobenzimidazole

A. V. Stetsenko and Yu. I. Bogodist

Ukrain. khim. zhur., 26, No. 1 (1960) 92-95

From quaternary salts of 1,2-dimethyl-4,5,7-trichlorobenzimidazole, one symmetrical and five asymmetrical cyanine dyes have been prepared, of formula—



(Z = 1,3-dimethyl-4,5,7-trichlorobenzimidazolyl; 3-methylbenzoxazolyl; 3-ethylbenzthiazolyl; 3-ethyl-6,7-benzobenzthiazolyl; 1-ethyl-quinoline; or 2-p-dimethylaminostyrene. Tabulated data give their absorption max. in ethanol (435-557 mμ.) and also the hypsochromic displacement of the absorption max. The hypsochromic displacement is expressed as difference of absorption max. of the asymmetrical dye and that of the average arithmetical value of the two corresponding symmetrical dyes. The magnitude of this hypsochromic displacement can be related to the basicity of the heterocycles contained in the asymmetrical dye; e.g. 2-methyl-4,5,7-trichlorobenzimidazole is found to be a stronger base than quinoline. G.J.K.

Vegetable Tannins. IV—Divi Divi Pods (C.I. Natural Brown 6)

K. N. S. Sastry, J. B. Rao, and Y. Nayudamma

Bull. Central Leather Research Inst., Madras, 6 (1960) 267-274

J. Amer. Leather Chem. Assoc., **55** (Aug 1960) 471-472

Chromatographic analysis of extracts with ethyl acetate, acetone, and methanol, respectively, showed presence of 3,6-digalloyl glucose, corilagin, and gallic, chebulinic, chebulagic and ellagic acids, with numerous unidentified compounds. C.O.C.

Chemical Constitution of the Melanins (C.I. Natural Brown 9)

M. Rangier

Compt. rend., **249** (1959) 1954-1955

Using Buchner's method (cf. H. Mason, *Chem. Abs.*, **45** (1951) 153) for preparing melanin, a yellow solution with black flocks is formed from aqueous solutions of adrenochrome at different pH by precipitation in air and washing with HCl. The reaction proceeds more rapidly as pH is lowered and temperature increased. Melanisation is slow at pH 6.0, O is consumed and a coloured film appears. Earlier (*Chem. Abs.*, **47** (1958) 5929) an unstable peroxide of structure in equilibrium with a quinone was described. Products resulting from hydration, which lose H₂O, are chain compounds analogous to silicones, which are converted by atmospheric O to chains having terminal COOH groups. The black compound has an anhydride structure; treatment with hot NH₃ yields an amide with an NH₄ salt, steam distillation of this in alkaline solution yields a product whose analysis (for C, H, total N and NH₃-N, O consumption) agrees well with the theoretical structure of a melanin. Melanins from hair and tumor tissue on infrared spectrographic analysis show approx. 7.3% N, as do the products obtained above. Comparisons of frequencies indicate presence of CO, COC, COO and CN groups in some melanins. The % of N indicates that natural melanins are derived from adrenalin; it would be larger if they were derived from dopa. C.O.C.

Chrome Yellows (C.I. Pigment Yellow 34)—Recent Developments—IV

A. J. Ryan and L. Williamson

Paint J. Australia New Zealand,**4** (6) (1959) 31, 33-35, 37-38*Chem. Abs.*, **54** (25 June 1960) 12611

Unstabilised Pb chromate made from pure Pb(NO₃)₂ darkens in a few days when exposed to sunlight. Stabilised chromes darken only after several months exposure. Presence of excess electrons or impurities in the crystal lattice is suspected. Coating the pigment particle with hydrous oxides of Group IV elements may improve colour retention by adsorption of the excess electrons. C.O.C.

Weather-resistant Zinc Sulphide Pigments (C.I. 77975)

W. Grassmann

Farbe u. Lack, **66** (1960) 67-74*Chem. Abs.*, **54** (25 June 1960) 12612

Photolytic formation of soluble ZnSO₄ is inversely proportional to the weather resistance of Lithopone (C.I. Pigment White 5) paints. The amount of photolysis was determined by solubility in acetic acid after a 4-hr. ultraviolet exposure of 1 g. ZnS in 250 c.c. water. Rain-water washings from painted exposure panels were collected and analysed for 14 months. All light-sensitive ZnS decomposed much more than light-stable ZnS. Addition of 1% of metals such as Ag, Al, As, Be, Bi, Co, Cd, Cr, Hg, In, Mn, Pb, Sb, Sn or V increases sensitivity to light and photolysis to 30-70%, while Fe, Co, Ni or Cu decrease it to 0-6%. With 0.5% Fe the most effective inhibition and least discoloration are obtained. It must be built into the crystal lattice as sulphide during manufacture. Presence of Cl is beneficial. Exposure tests of various paint systems containing stable ZnS (WZP) confirm their superiority after 4-yr. weathering. In linseed-oil paints combinations of ZnS and BaSO₄ with 15% white lead and 15% ZnO weathered well. The new ZnS also gave good results especially in colour stability in white polyvinyl alcohol paints. C.O.C.

Iron Pigments from Burnt Pyrites

J. Kriauciūnas

Lietuvos TSR Mokslu Akad. Darbai,*Ser. B* (2) (1958) 111-124 (Russian summary)*Chem. Abs.*, **54** (25 June 1960) 12612

Burnt pyrites is unsuitable as iron oxide pigment unless

residual S is removed. Purified pyrites can be used for making higher-quality pigments by treatment with H₂SO₄ at 80°C. Repeating the process twice results in 46% of the pyrites being converted to sulphate. Yellow pigments are obtained by treating 7% aq. FeSO₄ with Na₂CO₃ and oxidising the fresh precipitate by bubbling air through the dispersion. Brown pigments are obtained by treating 7% aq. FeSO₄ and borax with O in an autoclave at 180°C. The pigments obtained compare favourably in physical and chemical properties with those obtained by other methods. C.O.C.

Separation of Sulphur from Burnt Pyrites for Making Pigments

J. Kriauciūnas

Lietuvos TSR Mokslu Akad. Darbai,*Ser. B* (2) (1958) 101-110 (Russian summary)*Chem. Abs.*, **54** (25 June 1960) 12611

Burnt pyrites after removal of the S is useful for making pigments. Concentration of residual S decreased with decreasing particle size, only 1.04% S being present in the portion < 60 μ. separated from burnt pyrites of 2.73% S content. Sizing was done in a battery of 6 free-settling classifiers; the process was also effective in removing S and other impurities and in dissolving part of the sulphates. Three methods were used to remove residual S, (1) heating in an electric oven, at 900°C.—only half of the S was oxidised; (2) bubbling air through a column of an aqueous suspension of burnt pyrites—the rate of oxidation was slow; (3) oxidation in an autoclave at 160°C. and 4 atm. of air pressure above the water vapour pressure under those conditions of an aqueous suspension of burnt pyrites—complete oxidation was achieved in 4-5 hr. C.O.C.

PATENTS

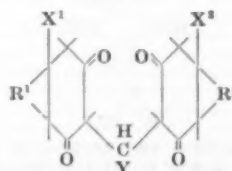
2,2'-Alkylenebis(1,3-cyclohexanediones) as Diazo-type Coupling Components

General Aniline

BP 839,948 (U.S.A. 16 July 1957)

USP 2,915,396 (16 July 1957)

Compounds of formula—



(R¹ and R² = Alk; X¹ and X² = H or Alk; Y = Alk, Ar, aralkyl, halogenophenyl, phenylpolyene or a heterocyclic ring), e.g. 2,2'-butylenedibis(5,5-dimethyl-1,3-cyclohexanedione) are good coupling components for producing red azo dye images. They have good stability and keeping qualities. C.O.C.

Orange Monoazo Pigments

FH

BP 844,394 (Germany 23 Mar 1956)

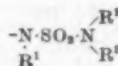
Orange monoazo pigments obtained by coupling diazotised 3-amino-4-chlorobenzamide with the anilide (or an o-alkoxyanilide) of 3-hydroxy-2-naphthoic acid are brighter and faster to light than those described in BP 728,601 (J.S.D.C., **71** (1955) 431) in which a di- or tri-substituted anilide of 3-hydroxy-2-naphthoic acid is the coupling component. E.S.

Metal(Chromium and Cobalt)-complex Monoazo Dyes for Wool, etc., having Aminosulphonylamino Groups

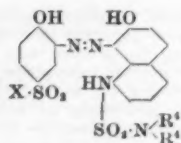
S

BP 840,178 (Switzerland 1 Feb 1957)

The title dyes contain 1 or 2 groups—



(R¹ = lower Alk or hydroxyalkyl; R² = lower Alk which may be subst. by Cl, OCH₃, or OC₂H₅; R³ = lower Alk which may be subst. by Cl, OCH₃, or OC₂H₅, or with N and R³ forms the radical of a saturated heterocyclic amine) and the 1:2 Cr or Co complexes of—

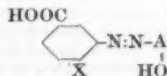


(X = Alk, especially CH_3 , NH_2 , NH-CH_3 , hydroxyalkyl-amino, or the radical of a heterocyclic amine bound to SO_2 through its N atom; $\text{R}^4 = \text{CH}_3$ or C_2H_5) are particularly useful. Thus diazotised 2-aminophenol-4-sulphonamide is coupled in presence of $\text{Ca}(\text{OH})_2$ with 1-dimethylaminosulphonylamino-7-naphthol. Heating with chromium ammonium sulphate in formamide gives the complex, which dyes wool grey from an acetic acid bath. E.S.

Metal(Chromium and Cobalt)-complex Monoazo Dyes for Wool, etc.

BASF BP 844,214 (Germany 5 May 1956)

Monoazo compounds—

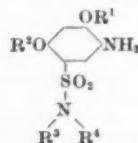


(X = OH or OAlk; A = residue of *o*-coupling azo component of the pyrazolone, quinoline, thionaphthene, or naphthol series) free from SO_2H groups are converted into 1:2 Cr- or Co-complexes to give the title dyes. If desired other suitable SO_2H -free monoazo compounds may be present. Thus 3-amino-4-hydroxybenzoic acid is diazotised and coupled with 1-(carboethoxy)amino-7-naphthol and the complex made by autoclaving at 130°C . with aq. chromium formate. It dyes wool blue from a weakly acid bath. E.S.

Metal(Chromium and Cobalt)-complex Monoazo Dyes for Wool

BASF BP 844,285 (Germany 9 Feb 1956)

Amines—

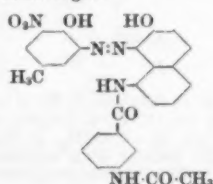


($\text{R}^1 = \text{H}$ or Alk; $\text{R}^2 = \text{Alk}$; $\text{R}^3, \text{R}^4 = \text{H}$, Alk, hydroxyalkyl, or phenyl, and R^3 and R^4 may belong to a common ring) are diazotised and coupled with *o*-coupling phenols, naphthols, pyrazolones, acetoacetylides, etc., and the products converted into 1:2 metal complexes which dye wool from a neutral or weakly acid bath, or into 1:1-complexes which may then be further treated with colourless complexing compounds as claimed in BP 692,073 (J.S.D.C., 69 (1953) 306). Thus 2,4-dimethoxyaniline-5-sulphonmethylaniline is diazotised and coupled with β -naphthol in presence of Na_2CO_3 . Heating at 130 – 135°C . with $\text{Na}_2\text{Cr}_2\text{O}_7$ in diethylene glycol gives a violet. E.S.

Metal(Chromium)-complex Monoazo Dyes for Wool from 1-Acylamino-7-naphthols

Gy BP 844,333 (Switzerland 29 Mar 1957)

The 1:2 Cr complexes of monoazo dyes free from COOH and SO_2H groups, and having 1-acylamino-7-naphthols as coupling components, have satisfactory solubility in water if the acyl group is the radical of a benzene-carboxylic or -sulphonic acid having in the benzene ring a low-mol.wt. acylated primary amino group. Thus 3-amino-4-hydroxy-5-nitrotoluene is diazotised and coupled with *N*-*m*-acetylaminobenzoyl-1-amino-7-naphthol in presence of aq. Na_2CO_3 and pyridine to give—

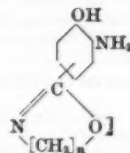


Boiling in aq. ethylene glycol monomethyl ether with sodium dialacylatochromate gives the complex, which dyes wool blue-grey from a neutral or weakly acid bath. E.S.

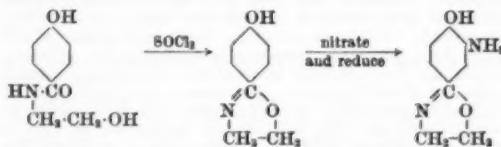
Metallisable Monoazo Dyes for Wool, etc.

S BP 841,930 (Switzerland 17 May 1957)

Amines—



($n = 2$ or 3) are diazotised and coupled with *o*-coupling hydroxy- or amino-compounds to give dyes which may be converted in substance or on the fibre into Co or Cr complexes. Thus 2-*m*-amino-*p*-hydroxyphenyloxazoline made by the route—

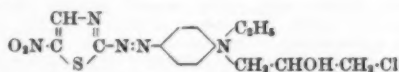


is diazotised and coupled in presence of Na_2CO_3 with 1-acetylamino-7-naphthol. It is a blue-grey metachrome or afterchrome dye. Heating with aq. CoSO_4 gives the 1:2 Co-complex which dyes wool and nylon grey. E.S.

Violet and Blue Monoazo Disperse Dyes having a Thiazole Residue

ICI BP 840,903 (4 Oct 1957)

2-Amino-5-nitro-thiazoles, which may carry substituents in the 4-position, are diazotised and coupled with *N*-3-chloro(or bromo)-2-hydroxypropylanilines to give dischargeable disperse violet and blue dyes for cellulose acetate, nylon, polyester, and polyacrylonitrile fibres. Thus 2-amino-5-nitrothiazole is diazotised in nitrosylsulphuric acid in presence of an acetic/propionic acid mixture and coupled with *N*-ethyl-*N*-3-chloro-2-hydroxypropylaniline giving—



which dyes cellulose acetate and nylon blue. E.S.

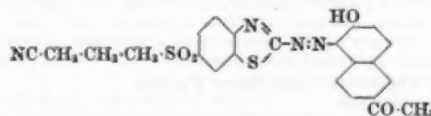
Metallisable Monoazo Disperse Dyes from 6-Substituted-2-aminobenzothiazoles

Eastman Kodak Co. USP 2,916,482 (15 Apr 1957)

2-Aminobenzothiazoles substituted in the 6-position by a group—



(X = S or SO_2 ; Y = CH_3CN , COOH , COOAlk , CO-NH_2 , CO-NHAlk , or CN ; Z = H or CH_3) are diazotised and coupled with 2-naphthol, which may have in the 6-position a halogen or CO-Alk substituent, to give disperse dyes which may be metallised, e.g. on cellulose acetate, with salts of Ni, Co, etc. Thus 2-amino-6-thiocyanobenzothiazole is refluxed with aq. Na_2S and γ -chlorobutyronitrile then added, to give the 6- γ -cyanopropylthio derivative. Oxidation with H_2O_2 in acetic acid then gives 2-amino-6- γ -cyanopropylsulphonylbenzothiazole, which is diazotised and coupled with 6-acetyl-2-naphthol to give—

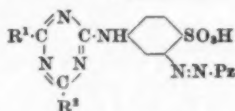


which dyes cellulose acetate red, converted to reddish blue by after-treating with $\text{Ni}(\text{SCN})_2$. E.S.

Yellow Monoazo Pyrazolone Dyes for Wool, etc., having a Cyanuric Residue

Ciba BP 840,742 (Switzerland 23 Jan 1956)

The title dyes are of the type—

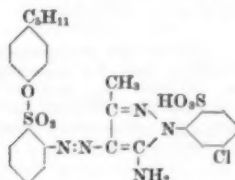


(R¹, R² = organic residues bound directly or preferably through N to the cyanuric residue and together contain preferably 6–12 C; Pz = residue of a 3-methyl-5-pyrazolone; > 2 SO₃H groups are present). Thus 1 mol. of cyanuric chloride is condensed with 1 mol. of aniline and 1 mol. of *m*-phenylenediamine-4-sulphonic acid. The product is then diazotised and coupled with 1 mol. of 1-*o*-chlorophenyl-3-methyl-5-pyrazolone, and the 3rd Cl atom of the cyanuric residue is then replaced by heating at 100°C. with methylamine in an autoclave, to give the greenish yellow acid dye. E.S.

Yellow Monoazo Pyrazole Acid Dyes

Gy BP 844,427 (Switzerland 23 Oct 1957)

Diazotised anilines having in the *ortho*-position a group SO₂R (R = a hydrocarbon, aryloxy, or organic amine radical) are coupled with sulphonic acids of 1-aryl-5-aminopyrazoles to give bright yellow acid dyes of good fastness to alkali and to light. Thus the *p*-amylphenyl ester of orthonic acid is diazotised and coupled with 1-(3'-chloro-6'-sulphophenyl)-3-methyl-5-aminopyrazole, giving—



which dyes wool greenish yellow from a bath containing (NH₄)₂SO₄. E.S.

Greenish-yellow Metal(Chromium)-complex Monoazo Dyes for Wool, etc.

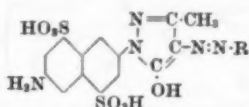
Gy BP 844,398 (Switzerland 5 June 1956)

The 1:2 Cr complexes of monoazo compounds free from SO₃H groups and obtained by coupling a diazotised 3-substituted anthranilic acid with a 5-pyrazolone give greener yellows than the similar dyes obtained from anthranilic acids unsubstituted in the 3-position. Suitable 3-substituents include Hal, Alk, Ar, OAlk, OAr, SAlk, SAR, CO-Alk, CO-Ar, NAlk, SO₂Alk, SO₂-Ar, SOAlk and SO-Ar. Thus the monoazo compound 3-chloroanthranilic acid → 3-methyl-1-phenyl-5-pyrazolone is heated in glycol with chromium acetate to give the complex, which dyes wool and nylon greenish yellow from neutral or weakly acid baths. E.S.

Metal(Copper)-complex Azo Direct Dyes from Naphthylpyrazolones

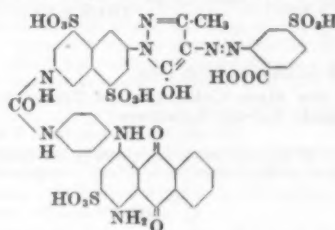
FBY BP 841,413 (Germany 10 Apr 1957)

Monoazo compounds—



(R = residue of a diazo compound having a cheating group *ortho* to the azo link) are N-acylated with, e.g. phosgene, fumaryl dichloride or cyanuric chloride, and the products are then made into Cu-complexes. Or phenyl chloroformate (I) may be used as the acylating agent and the urethanes so formed then react with amines, e.g. aminoazo and particularly aminoanthraquinone compounds, to give unsymmetrical ureas whose

Cu-complexes are direct dyes. Thus, using 4-sulphoanthranilic acid as the diazo component, N-acylating with I, and condensing with 1-*p*-aminoanilino-4-aminoanthraquinone-3-sulphonic acid there is obtained—

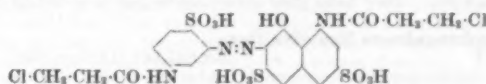


whose Cu-complex dyes cotton green. E.S.

Monoazo Reactive Dyes for Cellulose, having β-Halogenoethyl Groups

Ciba BP 845,567 (Switzerland 8 Mar 1957)

Anilinesulphonic acids having a substituent -NH-CO-CH₂CH₂-Hal are diazotised and coupled with suitable 1-acylamino-8-naphtholsulphonic acids (the acyl group may also carry a β-Hal atom) to give the title dyes, which react with cellulose under alkaline conditions. Alternative syntheses are available. Thus 4-β-chloropropionylaminoanthranilic acid is diazotised and coupled with *N*-β-chloropropionyl-H acid in presence of Na₂CO₃ to give—

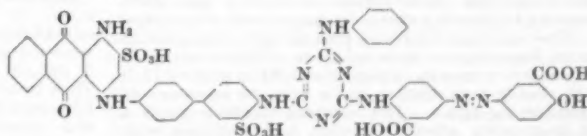


which dyes cotton red fast to washing by a process involving steaming in presence of aq. NaOH. E.S.

Bright Green Azo Anthraquinone Direct Dyes

S BP 844,353 (Switzerland 5 Aug 1957)

Bright green direct dyes made by combining a blue aminoanthraquinone dye and a yellow aminoazo dye by means of a cyanuric halide have improved solubility if the aminoazo dye has a COOH group *ortho* to the amino group. Thus the Cl in the monoazo compound 5-amino-2-chlorobenzoic acid → salicylic acid is replaced by NH₂ by autoclaving with aq. NH₃. The aminoazo compound so produced is condensed cold with 1 mol. of cyanuric chloride, and the product is then condensed at 60–65°C. with 1 mol. of 1-amino-4-[4'-(4'-amino)-phenyl]phenylaminoanthraquinone-2,3'-disulphonic acid. Finally the 3rd Cl of the cyanuric residue reacts with 1 mol. of aniline, giving—



which dyes cellulose bright green, becoming yellower and faster to washing when after-coppered. E.S.

Red Azoic Dye and Pigment

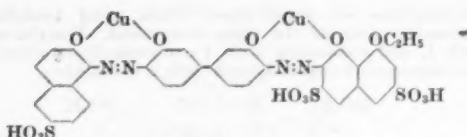
FH BP 840,804 (19 Nov 1956)

The azoic dye 2-ethyl-5-nitroaniline → 3-hydroxy-2-naphthol-β-naphthylamide, has better fastness to light and weathering on cellulose than the lower homologue from 2-methyl-5-nitroaniline. E.S.

Blue Metal(Copper)-complex Disazo Direct Dyes

FBY BP 845,491 (Germany 14 Aug 1957)

The title dyes are prepared by coppering disazo dyes made by coupling tetrazotised 3,3'-dihydroxy(or dialkoxy, or dichloro)benzidine with 1 mol. of a 1-alkoxy-8-naphthol-3,6-disulphonic acid and 1 mol. of an *o*-coupling naphtholmonosulphonic acid. They have good light fastness, especially after crease-resist finishing. Thus tetrazotised dianisidine is coupled first with an alkaline solution of 1-ethoxy-8-naphthol-3,6-disulphonic acid, and then with Schaeffer acid in presence of Na₂CO₃. Heating with ammoniacal copper sulphate then gives the copper-complex—

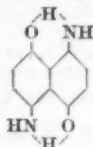


which dyes cellulose reddish blue.

E.S.

Dyes for the Mass Coloration of Fibres produced from Organic Solvent Solutions

BP 843,306 (Switzerland 5 Apr 1957)
Dyes produced by halogenation and/or hydrolysis and/or condensation with amines or hydroxy compounds—



or of alkylated or non-alkylated amino-1,4-naphthoquinones readily dissolve in organic solvents to give red, violet, blue or green solutions. Used for the mass coloration of cellulose acetate, polyacrylonitrile, polyethylene, etc., fibres they yield colorations of good to very good fastness to light, cross-dyeing, alkaline chlorination, oxalic acid, dry-cleaning, peroxide bleaching, and perspiration. They have good resistance to gas-fume fading.

C.O.C.

Anthraquinone Reactive Dyes

ICI BP 847,142 (13 Nov 1957)

Anthraquinone compounds substituted in < 2 α -positions by arylamino groups, at least one of which contains < 1 β -halogenoalkylsulphanyl group (I) and < 1 of the others contains < 1 SO_3H group, are water-soluble reactive dyes of excellent light and washing fastness for natural and synthetic fibres, especially cellulosic fibres. They are prepared by condensing β -halogenoalkylamines with anthraquinones containing α -aryl amino groups substituted by SO_3Cl groups (II), so that some II are converted to I and the remainder to SO_3H . Thus, 1,4-bis-(4'-phenylamino)anthraquinone-tetrasulphonyl chloride condensed at pH 7.5-8.0 with β -chloroethylamine hydrochloride gives a dye (containing approx. 2 SO_3H groups and 2 β -chloroethylsulphanyl groups per anthraquinone nucleus) which colours cellulosic fibres dull green of good fastness to light and washing.

A.T.P.

Fluorescent Brightening Agents

S. Pressner USP 2,915,472 (9 Jan 1956)

Water-insoluble dialkylamino coumarins are good fluorescent brightening agents for use on wool, silk, nylon, etc. They are very soluble in pine oil and are applied as aqueous dispersions of such solutions. Thus 4-methyl-7-diethylamino coumarin (1 part) dissolved in pine oil (2.7) can be readily emulsified in water with the assistance of an emulsifying agent. The resulting emulsion yields a good brightening effect even when highly diluted with water.

C.O.C.

Mixtures of Aminocoumarins having Alkyl and Aromatic Groups respectively in 4-position as Fluorescent Brightening Agents

BASF BP 840,605 (Germany 22 June 1957)

Mixtures of an aminocoumarin containing an aliphatic group in 4-position with one having an aromatic group in 4-position give much better brightening than either alone. It is possible to obtain a neutral white but by varying the proportions of the two components it is possible to obtain any tint from purest white to pale blue or green fluorescence. As the components exhaust at the same rate the mixture can be used in standing baths. An example of such a mixture is 70 parts of 4,6-dimethyl-7-ethylaminocoumarin and 30 parts of 4-phenyl-6-methyl-7-ethylaminocoumarin. This yields a neutral white on cellulose, cellulose ester, polyester, wool, nylon and acrylic fibres.

C.O.C.

Acid Pasting of Metal-free Phthalocyanine (C.I. 74100)

DuP USP 2,917,518 (14 July 1958)

The cost of the acid pasting process is immensely

reduced and a greener and brighter pigment obtained, if the pasting is done while preventing access of atmospheric oxygen to the acid pigment solution.

C.O.C.

Phthalocyanine Pigments

DuP USP 2,910,482 (8 Feb 1955)

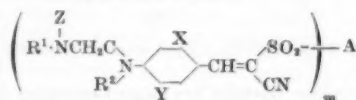
The title compounds (I) are Cu phthalocyanines containing in < 1 benzene nucleus, *meta* to the nearest CN group, halogen atoms (5.5-17% Cl or Br). They are obtained by reaction of finely ground CuCl_2 (II), urea (III) (15-40 mol. per mol. II), and a mixture of a phthalic epd. (acid, anhydride, amide, imide, NH_4 salt) (IV) and a 4-halogenophthalic epd. (V) (ca. 0.33-3 mol. V per mol. IV, IV and V together being 4 mol. per mol. II) at $\geq 190^\circ\text{C}$. in an inert organic solvent and in presence of a catalyst. I are in a previously unknown crystal phase, have high tinctorial strength, stability in organic solvents (no agglomeration in paints on standing), and are obtained directly in a pigmentary state (75-90 sq.m./g.) suitable for use in inks without further treatment. Thus, an aq. cake containing 4-chlorophthalic acid mono-Na salt (10) is heated at 150°C . with nitrobenzene (121) and 37% HCl (6-1) to remove H_2O . Phthalic anhydride (16-3), urea (61), micro-pulverised CuCl_2 (5-6) and NH_4 molybdate (0-07) are added at 110°C ., and the mass is stirred over 4-5 hr. to 175°C . and at $175-180^\circ\text{C}$. for 4-5 hr. It is cooled to 100°C ., drowned into Na_2CO_3 (17) in H_2O (50) and steam-distilled from nitrobenzene. After being filtered and washed, the cake is pasted 4-5 hr. at $85-90^\circ\text{C}$. in 22% H_2SO_4 (410), filtered, washed, dried at 60°C . in constant humidity, and screened. It has 5.8-7% Cl, is stable in boiling xylene for several hr. with no loss in tinctorial strength and no change in X-ray diffraction pattern. The loose agglomerates (0.5-3 μ) consist of primary crystals (0.03-0.50 μ) \times (0.02-0.25 μ) \times (0.01-0.04 μ).

A.T.P.

Styryl Dyes

S BP 843,643 (Switzerland 3 Aug 1956)

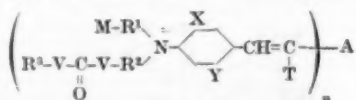
Strong, bright yellow dyes for mass-pigmenting oils, lacquers, plastics, cellulose sec. and tri-acetates, and polyamide and polyester fibres (during the melt-spinning of which most azo dyes decompose) where very good fastness is desired, have formula—



($\text{R}^1 = \text{Cl}$, Br , or $\text{R}^3\text{-CO-O}$, where $\text{R}^3 = \text{CH}_3$, CH_2Cl , C_2H_5 , C_6H_5 , n or *iso*- C_4H_9 , CH_3OCH_2 or $\text{CH}_2\text{OC}_2\text{H}_5$, $\text{R}^2 = \text{Alk}$ of 1-4 C, or where $\text{R}^1 = \text{R}^2\text{-CO-O}$, R^2 may be $\text{R}^3\text{-CO-O-CH(Z)-CH}_3$, $\text{X} = \text{CH}_3$ or C_6H_5 , $\text{Y} = \text{H}$, CH_3 , C_6H_5 , OCH_3 or OC_2H_5 , $\text{Z} = \text{H}$ or CH_3 , $m = 1$ or 2 , $\text{A} = \text{subst. or unsubst. benzene, naphthalene, tetrahydronaphthalene or diphenyl residue}$). They are obtained by condensing 1 mol. of an active methylene compound, $\text{A}(\text{SO}_2\text{CH}_2\text{CN})_m$ with m mol. of the requisite aldehyde. Thus technical 1-(*N*-ethyl-*N*-chloroethylamino)-3-methyl-4-benzaldehyde (22-5) is refluxed for 24 hr. in ethanol (30) and piperidine (1) with 4-methylbenzene-1-sulphonyl acetonitrile (19-5). The title compound where $m = 1$, $\text{R}^1 = \text{C}_6\text{H}_4\text{Cl}$, $\text{R}^2 = \text{C}_6\text{H}_5$, $\text{Z} = \text{Y} = \text{H}$, $\text{X} = \text{CH}_3$ and $\text{A} = 4$ -methylphenyl-1 crystallises on cooling. It colours polyethylene (from a xylene spinning dope) greenish yellow. 201 examples are given.

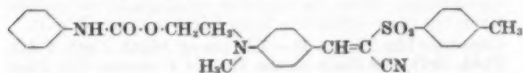
BP 843,644 (Switzerland 17 May 1956)

Related dyes to the above are—

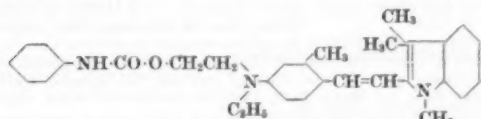


($\text{A} = \text{CN}$, carbalkoxy, carbalkoxyalkoxy, or a phenyl, diphenyl, naphthyl or tetrahydronaphthylmonosulphonyl radical which may contain Hal or Alk , or a 1,3,3-trimethylindoleninium (I) radical linked in the 2-position, or a naphthylene disulphonyl radical, $\text{X} = \text{H}$, CH_3 , C_6H_5 , OCH_3 or OC_2H_5 , $\text{Y} = \text{H}$, CH_3 , C_6H_5 , OCH_3 or OC_2H_5 , or where $\text{A} = \text{I}$, Y may be Hal or CF_3 , $\text{R}^1 = \text{alkylene of 1-4 C}$, $\text{R}^2 = \text{alkylene of 2-4 C}$, $\text{R}^3 = \text{subst. or unsubst. aliphatic, cycloaliphatic, or aromatic, free from}$

H₂O-solubilising groups, one V = H, other = NH, M = H, Alk, alkoxy, R²-V-CO-V, or where A = I, M may be CH₂CN, CHF₂, CF₃ or carbalkoxy, T = H when A = I and CN when A ≠ I, n = 1 or 2). They are prepared in a similar manner to the dyes of BP 843,643 and have similar uses. They can also be used as greenish yellow to red disperse dyes of excellent fastness to washing, sublimation, gas-fumes and light (BP 843,645 also refers to their use in melt-spinning of polyamides, polyesters and polythene). Dyes where T = H and A = I have outstanding affinity for Orlon. Thus, cellulose triacetate is mass coloured a fast bright greenish yellow with—



and Orlon is dyed a brilliant red of excellent fastness by—



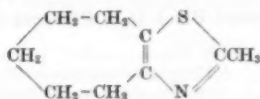
164 examples are given.

A.T.P.

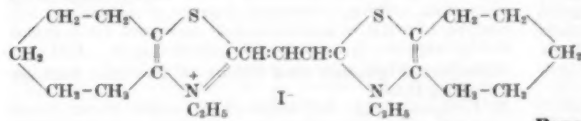
Thiazole Base and Cyanine Dyes containing a 5,6,7,8-Tetrahydro-4-cycloheptathiazole Nucleus

Sperry Rand Corpn. USP 2,916,487 (22 Aug 1955)

Treating the enol form of 2-chlorocycloheptanone with thioacetamide yields 2-methyl-5,6,7,8-tetrahydro-4-cycloheptathiazole (I)—



It is used as an intermediate for cyanine dyes. Thus its ethiodide refluxed with diethoxymethyl acetate in presence of pyridine and triethylamine yields the reddish purple (in methanol) dye—

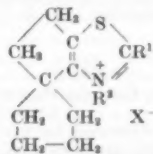


C.O.C.

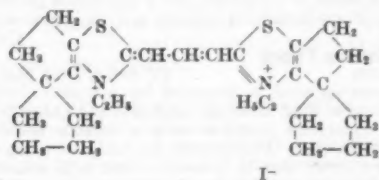
Cyanine Dyes containing a Spiro (4.4) nonano (1,2d) Thiazole Nucleus

Sperry Rand Corpn. USP 2,916,488 (27 June 1956)

Compounds of formula—



(R¹ = CH₃, C₂H₅ or n-propyl; R² = Alk or aralkyl; X = anion) are used as intermediates for cyanine dyes. Thus refluxing a pyridine solution of 2-methylspiro (4.4) nonano(1,2d) thiazole ethiodide, diethoxymethyl acetate and triethylamine yielded the reddish blue (in methanol) dye—



C.O.C.

β-Carotene (C.I. 75130) Compositions for Colouring Butter and other Foodstuffs

Hoffmann-La Roche USP 2,916,385 (8 Oct 1957)

Finely divided carotene is dispersed in hot edible saturated aliphatic acid of 16–22°C. On being cooled this yields a semi-solid gel which on being stirred or agitated vigorously becomes a free-flowing, viscous liquid. If this liquid is heated and cooled it again forms a semi-solid gel. The product is readily incorporated into butter at any stage of its manufacture, e.g. to the cream before pasteurising or to the butter when the salt is added.

C.O.C.

Carbon Black (C.I. Pigment Black 6 and 7)

Godfrey L. Cabot USP 2,917,370 (26 Apr 1950)

Describes production of Oil Blacks of quality at least equal to that of Gas Blacks.

C.O.C.

Carbon Black (C.I. Pigment Black 6 and 7)

Columbian Carbon Co. USP 2,915,371 (10 June 1955)

Pigment of low oil-absorption properties and of average particle size much less than that of existing thermal blacks is obtained by dispersing hydrocarbons in an inert gas at a temperature above that at which the hydrocarbons are decomposed to Carbon Black. The mixture is passed continuously through an externally heated chamber whose walls are kept at a temperature below that of the hydrocarbon suspension. A good yield of a product of exceptionally uniform particle size is obtained.

USP 2,915,372 (11 June 1957)

The characteristics of the pigment obtained from the same liquid hydrocarbon by the process of USP 2,440,424 (J.S.D.C., 64 (1948) 412) can be modified by variations in the process and especially by the manner in which the make sprays are injected into the reaction chamber.

C.O.C.

Furnace Black (C.I. Pigment Black 7)

Phillips Petroleum Co. BP 840,336

Process and apparatus which produces a good rate of yield of a pigment which imparts both higher abrasion resistance and lower heat build-up to rubber mixtures in which it is incorporated.

C.O.C.

Graphite (C.I. Pigment Black 10) from Polynuclear Aromatic Hydrocarbons

Union Carbide Corpn. USP 2,915,370 (3 Oct 1955)

Treating the volatile polynuclear hydrocarbons present in coal tar pitch with resin-forming agents, e.g. formaldehyde with ZnCl₂ as catalyst, yields resins which can be coked, calcined and graphitised to yield a soft and highly crystalline synthetic graphite.

C.O.C.

Beneficiated Hydrous Iron Oxide Pigment (C.I. 77492)

DuP USP 2,917,400 (15 Apr 1957)

Water-wet hydrous iron oxide of average particle size 16–100 mμ. and containing particles of a size capable of scattering light is beneficiated by treatment with a hydrophobic aliphatic monocarboxylic acid in presence of a volatile water-immiscible organic solvent for the acid. This gives the pigment a hydrophobic surface bound by salt linkages. The free water is removed and the resulting dispersion centrifuged at 6000–7000 g. This removes the particles capable of scattering light and leaves a dispersion in the organic liquid of particle size incapable of scattering light. Coating compositions pigmented with this dispersion have superior transparency, brilliance, two-tone effect and jetness of mass tone compared with those pigmented with ordinary hydrous iron oxide.

C.O.C.

Rendering Dyes and similar Powders Non-dusting

ICI BP 842,791 (31 Mar 1958)

The dye or powder is mixed with 0.1–2.0% by weight of a polar organic liquid of viscosity < 50 cp. at 20°C. The liquid is of 15–30°C and consists of an alkenyl, Ar or subst. Ar, ether oxygen, thioether, mercapto, carbonyl, cyano, N-subst. carbamyl, amino or subst. amino, or sulphonyl group linked to sufficient straight or branched chain Alk each of 4–18 C to satisfy the valencies of the polar group, e.g. octadecene, dodecyl benzene, condensate of naphthalene sulphonic acid and formaldehyde, and 2,4-dichloro-6-(2'-chloroanilino)-s-triazine.

C.O.C.

Aqueous Dispersions for Coating Fibrous Materials

Keiner & Co. BP 842,646 (4 Feb 1957)

A suspension of a pigment and/or filler in an aqueous

solution of a Na, K, NH₄ or amine salt of carboxyl groups or vinyl-group polymers or copolymers is used. Thus a viscous solution of ammonium polymethacrylate is mixed with a pigment pulp to provide a product which, diluted with water, can be used for colouring leather in a drum dyeing machine. C.O.C.

Pelleting of Carbon Black (C.I. Pigment Black 6 and 7)
(I p. 633)

Titanium Pigments and Effect of Particle Size on Printing Inks (V this page)

Heat-fastness of Disperse Dyes on Synthetic Fibres (VIII p. 647)

Dichroism of Dyes in Stretched PVA Sheet. III—Direction of Absorption of Pincyanol Iodide (VIII p. 647)

Aqueous Dispersions of Diazo Components and of their Mixtures with Coupling Components (VIII p. 647)

Dyeing Polyolefin or Polyvinylidene Chloride Filaments with Monoazo Disperse Dyes (VIII p. 648)

Improving the Dyeing Properties of Polymers Containing Nitrile Groups (VIII p. 648)

Indole Derivatives for Rendering Natural and Synthetic Polymers Self-sterilising and Bactericidal (X p. 651)

Photoreduction and Photo-oxidation Sensitised by Dyes (XIV p. 655)

Factors affecting the Intensity of J-bands in the Absorption Spectrum of Water-Gelatin Solutions of Dibenzo-thiacarboyanine Dyes (XIV p. 655)

Centrifugally Accelerated Paper Chromatography of Chloroplast Pigments (XIV p. 655)

Effect of Dye Concentration on Light Fastness of Dyed Fibres (XIV p. 656)

V—PAINTS; ENAMELS; INKS

New Pigment-mixture Data

E. Friel *J. Opt. Soc. Amer.*, **50** (Aug 1960) 791-801
Using different positions for some pigments at different lightnesses when preparing a diagram of the type previously described (*J. Opt. Soc. Amer.*, **49** (Dec 1959) 1159-1168; J.S.D.C., **76** (Apr 1960) 249) enables information about the colours resulting from mixture of the pigments at these points to be summarised very well in graphic form. A method of assembling the pigment positions for lightnesses 9, 7, 5, 3 and 1 of a 10-step scale is described. C.O.C.

Electron Microscopy and Paint Technology—A Survey

S. H. Bell
J. Oil & Col. Chem. Assoc., **43** (July 1960) 466-492

Survey of 15 years' work, conducted mainly at the Paint Research Station. Investigation of pigments, paints in depth, paint surfaces, pigments in media, emulsion paints, is described. There is a brief reference to the microscopy of organic materials, and the application of reflection microscopy and electron interference methods. There are 32 electron micrographs. J.W.D.

Luminescent Paints

M. Dérivé *Peintures, pigments, vernis*, **36** (1960) 76-82
Discussion of modern theories of fluorescence and phosphorescence is followed by a review of properties and behaviour of the most important luminescent pigments of the zinc sulphide group activated with various metals, as well as the alkaline-earth sulphide pigments prepared with different fluxes. The formulation of paints is discussed, with particular reference to the chemical constitution of the binders required for each type to obtain and maintain maximum luminescence. C.O.C.

Chemical Nature of Paint Film Surfaces

W. T. M. Johnson *Off. Dig. Fed. Soc. Paint Technology*, **32** (Aug 1960 Part I) 1067-1078

Paint surface is lightly abraded with KBr powder, using a fine steel-wool pad; apparatus is described with the aid of four photographs. The KBr powder, contaminated with abraded surface material, is collected by means of a vacuum cleaning device and is pressed into a KBr disc which is then used to yield an infrared spectrum; this

discloses the chemical composition of the surface material. By this means the composition of the upper 50 Å. of film surface can be studied. Results indicate that film surfaces differ markedly from the bulk of the film. In a paint containing a silicone oil, the silicone concentrated at the film surface, the bulk of the film showing no trace. Similarly, an amide slip-agent was found to be present exclusively in the surface layer of a polyethylene film. In a paint film, the pigment was found under a clear layer of binder at the surface. A paint containing a plasticiser showed a higher plasticiser concentration at the film surface than within the film. No evidence has yet been found that abrasion alters the chemical condition of the surface layer, but the possibility is being borne in mind. J.W.D.

Chrome-Tin Pink. II—Effects of MgO, ZnO, CdO, PbO, SrO and BaO on the Hue of Chrome-Tin Pink
E. Kato, H. Takashima, and S. Kanaoka

Nagoya Kōgyō Gijutsu Shikensei Kōkoku, **9** (1960) 147-151

Chem. Abs., **54** (25 June 1960) 12611

Chrome-Tin Pink is Sn sphene stained by a little Cr. The hues of many varieties of Chrome-Tin Pinks, prepared by replacing part of the CaO of normal Sn sphene, CaO-SnO₂-SiO₂ by MgO, ZnO, CdO, PbO, SrO or BaO, were studied by spectral reflectance and I.C.I. colour specification. The shape and site of the concave of each reflectance curve, which is at about 520 mμ, and determines the hue of Chrome-Tin Pink, were hardly affected by the RO composition of Sn sphene. The hue was reddish purple. Chrome-Tin Pinks containing MgO, ZnO or CdO were bluer as a result of higher reflectance in the short-wave region. Those containing PbO, SrO or BaO were redder because of lower reflectance in the short-wave region. C.O.C.

Modified Channel Black (C.I. Pigment Black 7) for Printing Ink

A. Ya. Korolev, T. N. Malova, and V. I. Romanova

Poligraf. Proizvodstvo (7) (1957) 25-27

Chem. Abs., **54** (25 June 1960) 12612

Oxidation of Channel Black in water in presence of a catalyst at room temperature increases the surface polarity of the pigment particles. This results in lower oil capacity and viscosity and increased affinity for binder. The ink has greater colouring power and good printing properties. With increasing degree of oxidation, the content of volatile matter and O increased by 3-6 and 2-5%, respectively, and the pH decreased by 2. C.O.C.

Titanium Pigments and Effect of Particle Size on Printing Inks

H. C. Spreen *Am. Ink Maker*, **38** (1) (1960) 32-33, 35-38

Chem. Abs., **54** (25 June 1960) 12612

The critical factors influencing the hiding power of Ti pigments in printing-ink films are the particle size of the particles and the distance between them. Maximum hiding power in sq.ft./solid gal. is reached at 30% pigment volume concentration. For maximum efficiency, the particle size of the extenders must be in the same range as that of the pigment. C.O.C.

PATENTS

Colorants for Inks, Carbon Papers and the like

Caribonum *BP* 842,655 (16 May 1957)

Writing or prints obtained by use of a colorant and finely divided antimony are very suitable for being copied by thermo-sensitive copying processes. Use of antimony has the advantage over use of graphite in that much smaller quantities are needed. C.O.C.

Ink for Use on Chlorinated Plastics

Minnesota Mining & Mfg. Co.

USP 2,915,416 (16 Sept 1953)

An ink made of a pigment and a liquefiable polymer of trifluorochloroethylene is applied and then heat-set. C.O.C.

Film Carbon Paper

Caribonum *BP* 840,673 (29 Mar 1957)

The coating used is imparted by a fluid composition comprising a fluid colorant and a vinyl chloride-vinyl acetate copolymer together with a volatile solvent for both components. Plasticisers, insoluble solid pigments, fillers, etc., may also be present. Oleic acid solutions of solvent dyes are suitable fluid colorants. C.O.C.

Weather-resistant Zinc Sulphide Pigments (C.I. 77075) (IV p. 639)

New Developments in the Soluble Vinyl Aromatic-Diene Copolymer Paint Resin Field (XIII p. 654)

VI—FIBRES; YARNS; FABRICS

Stiffness of Woven Textiles

D. N. E. Cooper

J. Textile Inst., **51** (Aug 1960) T 317-T 335
Fibre stiffness depends upon fibre substance, cross-sectional shape and denier. Yarn stiffness is usually controlled by fibre stiffness, twist having little effect under normal conditions of lubrication. When yarn is woven into fabric, a wide range of stiffness is possible, depending upon the extent to which free movement of fibres is inhibited by fabric structure or finish. Fabric stiffness may vary with variation in the direction of bending but for most purposes measurements along warp, weft and one other direction suffice to describe it. C.O.C.

Properties of Cotton containing Radiation-polymerised Acrylonitrile

J. C. Arthur and R. J. Demint

Text. Research J., **30** (July 1960) 505-509
Relatively large amounts of acrylonitrile in aqueous $ZnCl_2$ were applied to cotton and then radiation-polymerised. Microscopic examination showed that the polymer was inside the fibre. There was only slight decrease in breaking strength, significant increase in elongation at break and decreased fibre stiffness. Resistance to acid and to heat was comparable with that of untreated cotton. C.O.C.

Unit Cell of Cellulose Triacetate (Variety I)

J. Toussaint and B. Passagez

Bull. classe sci., Acad. roy. Belg. (5) **45** (1959) 169-174
Chem. Abs., **54** (25 June 1960) 12574
Cellulose triacetate molecules heated above 200°C. are oriented in parallel chains, designated as "structure I". In "structure II", the cellulose chains cross each other. C.O.C.

Propagation of Excitation Energy in Keratin

A. R. Haly

Trans. Faraday Soc., **56** (Aug 1960) 1252-1259
Effects of ultraviolet radiation on a wool fibre are found in the near-axis of the fibre. It would be expected that this region would be protected by its sheath of protein material. There appears, therefore, to be a transfer of excitation energy over relatively large distances. It is concluded that this is not due to electron or proton migration but may result from diffusion of photochemical products or more direct transfer of excitation energy by means of exciton diffusion or sensitised fluorescence. W.R.M.

Kinetic Studies of Wool-Water System. I—Influence of Water Concentration

I. C. Watt *Text. Research J.*, **30** (June 1960) 443-450

Studies were made by following weight changes of fibres after changes of external water vapour pressure. Sorption kinetics were found to be dependent on both change in vapour pressure and initial regain of the wool. For low regain, sorption is Fickian with a concentration-dependent diffusion coefficient. For higher regain, part of the absorption takes place by a non-Fickian mechanism; the relative contributions of Fickian and non-Fickian mechanisms to total uptake vary with initial regain. S.B.D.

Polymerisation within Wool Fibres

A. Schöberl and D. Wagner

Melliand Textilber., **41** (Aug 1960) 984-988
Experiments were carried out using acrylonitrile and methyl methacrylate. Microscopical inspection shows that polymerisation occurs inside the fibre. It is assumed that the polymer is partly chemically bound to the fibre. A reaction mechanism involving the -S-S- linkage is suggested. W.M.

Reactivity of the Disulphide Bond in Wool

L. J. Wolfram and J. B. Speakman

Nature, **187** (13 Aug 1960) 595-596
Evidence is given confirming that formation of intermediate oxidation products, e.g. the sulphone, is sufficient to impair the setting power of wool. C.O.C.

Action of Tetrakis(hydroxymethyl)phosphonium Chloride on Wool

L. S. Bajpai, C. S. Whewell, and J. M. Woodhouse

Nature, **187** (13 Aug 1960) 602-603
Wool treated with tetrakis(hydroxymethyl)phosphonium chloride absorbs more acid dye but less basic dye than untreated wool. Addition of tetrakis(hydroxymethyl)phosphonium chloride to the dyebath greatly accelerates the rate of dye absorption. Stretched wool boiled in a solution of the above compound and then relaxed for 1 hr. in boiling water retains more set than wool which has been set by boiling in water alone. Probably the effect of the treatment is primarily to reduce the disulphide bond, and this is accompanied by absorption of phosphonium ions and so increases the number of positive sites in the fibre. C.O.C.

Wool Dyeing and Finishing in the West of England

K. G. Ponting *Dyer*, **124** (26 Aug 1960) 301-306

An illustrated historical account of woollen cloth trade. Bristol was original centre but trade declined, passing to nearby villages, ca. 14-15th centuries, because city guilds would not use mechanised fulling by water power. Gild dyeing regulations specified woad—with alum. Principal export of Tudor period was undyed, fulled, tented broad cloth—this ceased in 17th century and dyed wool cloth trade was renewed. Indigo replaced woad, the Stroud scarlets were cochineal, madder was used for ordinary reds and browns, weld for yellows, logwood for blacks. Pattern books, 200 years old, show wide range of colours obtained with these few dyes. It was mostly piece dyeing (except navy-dyed indigo and Medley mixtures) on fulled cloth, and by primitive methods. Finishing was important, plain weaves were given the unrivalled doeskin handle by heavy fulling and sequences of raising and cutting (the shearmen led riots against early mechanisation; the circular cutting machine, and rotary milling machine were both West Country inventions). In spite of Yorkshire's prominence, the West retained broad cloth trade, based on finishing technique. Nowadays the most modern dyeing machinery is used and more high-quality cloth is produced than ever. There are three centres—Stroud for traditional billiard cloths, etc.; Trowbridge for men's suitings and new fibres; and Somerset for woollens and worsteds. R.A.P.

Microscopic Studies of the Swelling of Chemically and Physically Modified Polyamide Fibres

W. Bobeth

Faserforsch. und Textiltech., **11** (July 1960) 305-311
Swelling in sulphuric acid is used to study the effects of heat, ultraviolet light, different setting treatments, and dyeing and bleaching processes. The influence of these and some chemical treatments on swelling times is discussed. W.R.M.

Intrinsic Viscosity and Molecular Weight of Polyethylene Terephthalate

N. G. Gaylord and S. Rosenbaum

J. Polymer Sci., **39** (Sept 1959) 545-547
A comparison of molecular weight data from end-group determination and viscosity measurements. P.T.S.

Strength and Properties of Glass Fibres

W. F. Thomas *Phys. and Chem. Glasses*, **1** (1960) 4-18

Chem. Abs., **54** (25 June 1960) 12521
The tensile strength of glass fibres is independent of fibre diameter. Annealing, even at < 100°C., causes decrease in strength. Breaking stress decreases with increase in R.H. and with increase in time of exposure to humidity. HCl vapour immediately decreases strength to 80% of the original, but longer exposure causes no further change. Immersion in aq. HCl of up to 0.01 N. does not affect strength, but stronger solutions decrease the fibre strength rapidly. Brief immersion in CCl_4 , acetone, benzene, toluene, or xylene has no effect on fibre strength. Fibres immersed in a solution of a silicone and then exposed to air are not weakened unless the silicone contains Cl. C.O.C.

PATENTS

Mass-coloured Regenerated Cellulose Fibres

Algemene Kunstzijde Unie

BP 843,243 (U.S.A. 24 May 1957)
Cellulose solution for fibre manufacture has dispersed in it an organic pigment and a white inorganic pigment, the

proportion of the latter being sufficiently high to cause filaments produced from the solution to show only one colour when viewed from different angles but not so high as to cause delustring. C.O.C.

Amylose Fibres

Kalle & Co. BP 847,431 (Germany 13 Apr 1957)
Amylose base-material, i.e. either pure amylose or starch fractions containing > 50% of amylose, is dissolved in aqueous alkali and then extruded into an aqueous acidic solution. The amylose must have average mol.wt. < 20,000 (by Potter and Hasnid's method, *J. Amer. Chem. Soc.*, **73** (1951) 593). The product can be used as a scaffolding thread in the manufacture of textiles. It is dissolved by amylases and so can be removed when the textile is being desized. C.O.C.

Delustred or Pigmented Polyamides

Vereinigte Glanzstoff-Fabrik

BP 842,675 (Germany 8 May 1957)
Much more uniform dispersion of delustrant or pigment added to the monomer is obtained if there is present 0.5-30.0% on the weight of the delustrant or pigment of aluminium phosphate. C.O.C.

Pigmented Polyesters

N.V. Onderzoekingsinstituut Research

BP 840,215 (Holland 19 Oct 1957)
Homogeneous pigmentation of polyesters is obtained if the pigmentation is done in presence of a compound of formula R^1-X-R^2 (R^1 = hydrophobic group whose hydrophobicity at least equals that of the group $CH_3(CH_2)_6$; R^2 = hydrophilic group containing a group of formula $-(CH_2CH_2O)_{3-40}$; X = a group linking R^1 and R^2 by primary valencies), e.g. *p*-octylphenylheptaethylene glycol. C.O.C.

Stretch Yarns

Deering Milliken Research Corp.

BP 842,703 (U.S.A. 14 July 1955)
A thermoplastic yarn of high molecular orientation is stretched beyond its elastic limit at a temperature and tension to give it maximum modulus of elasticity and decreased characteristic elongation to break. It is then passed over a sharp edge at an acute angle. C.O.C.

Dyes for the Mass Coloration of Fibres produced from Organic Solvent Solutions (IV p. 642)

Chitin Fibres and Chitosan Printing (IX p. 648)

Reaction of Dimethylol Urea with Cotton (X p. 649)

Reaction of Dimethylol Ethylene Urea with Cotton (X p. 649)

Application of U.V. Absorbers to Terylene (X p. 650)

Reaction between Polyvinyl Alcohol Fibre and NN' -Methylenebisacrylamide (X p. 650)

Bactericidal Synthetic and Proteinaceous Fibres by Use of Neomycin (X p. 651)

Anti-static Finish (X p. 651)

Improving Shaped Products Containing Carbonamide Groups. Anti-static Finish (X p. 651)

Removing Discoloration Caused by Sulphur in Viscose Rayon (X p. 652)

Reducing the Swelling Value and Improving the Stability of Crimp of Regenerated Cellulose Fibres (X p. 652)

Reducing the Tendency of Spun Polyethylene Terephthalate Fibres to Pill (X p. 652)

Treating Polymers Containing Nitrile Groups with Hydroxylamine (X p. 652)

Modifying Synthetic Condensation Polymers (X p. 652)

Mineral Fibres Coated with Synthetic Resins (X p. 652)

X-ray Studies on Polyvinyl Alcohol Fibre (XIV p. 656)

Determination of the Continued-tear Resistance of Fabrics (XIV p. 656)

Behaviour of Sample Pleated Strips of Wool Fabric in an Extensometer (XIV p. 656)

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

Capillary Processes in Wet Treatment of Fabrics

A. N. Braslavskii and A. M. Motorin

Zhur. priklad. khim., **33** (June 1960) 1391-1396
Change in capillarity of cotton fabrics in wet processing (mercerisation, printing, etc.), using water and ethanol,

has been investigated, and a relation between the capillary-porous structure of the fabric and the kinetics of penetration by capillary processes established. G.J.K.

Improvements in Chlorite Bleaching

R. H. Parkinson

J.S.D.C., **76** (Sept 1960) 552

Fluorescent Brightening Agents and Blueing Agents in Wool Processing

AATCC Delaware Valley Section

Amer. Dyestuff Rep., **49** (8 Aug 1960) 565-584

The wide range of fluorescent brightening and blueing agents available for use on loose wool has been evaluated for efficiency and fastness to ordinary wool processing and the results are given in tables and photographs. Work on their effect on dyeing has shown that transfer of fluorescent brightening agents from the wool to synthetic fibres blended with it is a serious cause of streakiness and uneven dyeings. Fadeable dirt can be compensated for to a certain extent. Presence of fair quantities of fluorescent brightening agents can be detected under ultraviolet radiation and of blueing agents in daylight but determination of smaller amounts is complicated by the natural fluorescence of wool and presence of other extractable matter. Combinations of the two types of agents give the most satisfactory results under varying lighting conditions. Generally, significantly improved wool colour showed the greater loss of whiteness in subsequent processing. Application in continuous processing demands careful attention and whitened wools can cause difficulties particularly in matching, in fastness to light of pale dyeings, and in migration to synthetic fibres in blends. Unless a wool is to be left in its natural colour in the final product, the improved appearance may lead to difficulties in subsequent processing without doing anything which the dyer cannot do possibly better. C.O.C.

Studies of Dry-cleaning. III—Dry-cleaning Studied Microscopically

R. Mönch

Faserforsch. und Textiltech., **11** (July 1960) 327-333

The dry-cleaning process is studied microscopically using substances soluble and insoluble in water as dirt. Wool and cellulose fibres are more easily dry-cleaned than synthetic fibres. Microphotographs are shown and the observed processes discussed. W.R.M.

Bleaching Compositions (III p. 634)

VIII—DYEING

Principles of Padding

R. A. Peel

Dyer, **124** (15 July 1960) 95-96

Brief bibliography of padding for apprentices; six rules for good work and a description of the lever mangle.

R.A.P.

A Contribution to the Physical Chemistry of Dyeing

W. Luck

Melliand Textilber., **41** (Mar 1960) 315-320

The dyeing process is governed by several interconnected equilibria. Quantitative measurement by spectroscopic studies is described and the effect of non-ionic auxiliaries on dye association and on level dyeing is discussed in detail. W.M.

Incorporation of Ultraviolet Absorbers in Textiles

D. J. Delano

Amer. Dyestuff Rep., **49** (25 July 1960) 536-537

Improved fastness to light can be obtained by use of ultraviolet absorbers. The best field of use is with dyes of fairly good light fastness, as with the increased fastness to light given by the ultraviolet absorbers such dyes would be suitable for goods demanding maximum fastness to light. C.O.C.

Adsorption of Dye Mixtures by Cellophane Sheet. II—Quantitative Discussion of Adsorption of Dyes

Y. Horiki

Bull. Chem. Soc. Japan, **33** (July 1960) 974-979

The single dyeings of Chlorazol Sky Blue FF (C.I. 24410) and Chrysophenine G (C.I. 24895) and their mixture dyeing at 50°, 70° and 90°C. are described in detail and, for the former dyeings, the surface layer volume in Vickerstaff's equation (cf. *Proc. Roy. Soc.*, **A 192** (1948) 292) is discussed to obtain constant values of the standard affinity. In the mixture dyeing, the behaviour of the dyes

may be well explained quantitatively on the basis of the information of the single dyeing and the interaction of both dyes in the dyebath. H.H.H.

Effect of Solvents in Wool Dyeing

L. Peters, C. B. Stevens, J. Budding, B. C. Burdett, and J. A. W. Sykes *J.S.D.C.*, 76 (Sept 1960) 543-550

Uneven Dyeing of Sheep and Lamb Fur Skins Fixed by Formaldehyde

J. Jancso *Kozarstvi*, 9 (1959) 363
J. Amer. Leather Chem. Assoc., 55 (Aug 1960) 468-469

Fixation with formaldehyde (HCHO) lowers the affinity of the fibres for oxidation bases. Uneven dyeing is usually caused by uneven ironing, poor control of temperature, and especially by uneven brushing on of the HCHO. Oiling is a frequent cause of uneven dyeing; soap causes stains on brown dyeings and sulphated oils on browns and greys. Mineral and vegetable oils and non-ionic detergents have no effect. Red stains on brown dyeings and violet stains on greys can be caused by the furs being left in damp sawdust. Stains caused by chemical variations in the wool can be much reduced by stronger killing. Urine stains can be lessened by bleaching with H_2O_2 and $Na_2S_2O_4$ before or after tanning; this increases the number of skins suitable for pastel dyeing by 30-40%, but bleaching with H_2O_2 alone only gives an increase of 15-20%. Bleaching during killing is not effective. C.O.C.

Use of Pyrocatechol (C.I. Oxidation Base 26) in Fur Dyeing

—, Eckhart *Kozarstvi*, 9 (1959) 364
J. Amer. Leather Chem. Assoc., 55 (Aug 1960) 469-470

Pyrocatechol gives better results with oxidation bases than does phenol. A deep black is obtained with 2 g./l. each of *p*-phenylene diamine (C.I. Oxidation Base 10) and pyrocatechol, the depth of the dyeing being equal to that obtained with 6 g./l. *p*-phenylenediamine alone. Pyrocatechol is useful for obtaining browns only if the oxidation base contains NO_2 groups. For blacks mordanting requires 3-4 g. $K_2Cr_2O_7$ /l. but for greys > 2 g./l. should be used. Where pyrocatechol is used, 2 g./l. of H_2O_2 suffices for oxidation. Blacks and greys obtained with help of pyrocatechol have impaired fastness to light and ironing. Cu salts are not suitable as mordants with pyrocatechol. A grey can be obtained by mordanting with $FeSO_4$ and then dyeing with *p*-phenylene diamine and pyrocatechol. Mordanting is best done with $K_2Cr_2O_7$, but strict control of pH is necessary to prevent over-mordanting of bristle hairs. Dyeing with pyrocatechol is best done at 40-45°C. The time needed for drumming in sawdust is shortened. C.O.C.

Even Dyeing of Furs

F. Brugger and L. Paris *Kozarstvi*, 9 (1959) 363
J. Amer. Leather Chem. Assoc., 55 (Aug 1960) 469

When sheep or lamb fur skins with wool fixed by HCHO are dyed, the tips are usually lighter than the bases. Fixing with HCHO slows down the rate of mordanting with $K_2Cr_2O_7$, but the final amount of Cr_2O_3 taken up is increased. HCHO-fixed wool absorbs more alkali during killing. The tips dye lighter because additional bonds are formed in the wool by the HCHO treatment and this causes oxidation bases to diffuse more slowly into it. Diffusion is accelerated by increase in temperature, but as this also causes increase in mol.wt. of the dye, oxidation must be slowed down. Adding the H_2O_2 to the dyebath only after the furs have been in the bath for 2 hr. and then raising the temperature results in even dyeing from tip to root of the fur and saves 20-25% of dye. C.O.C.

Heat-fastness of Disperse Dyes on Synthetic Fibres

J.-P. Merminod *Teintex*, 25 (15 Aug 1960) 551-563

A table of proposed tests is given from which several were selected for trial including the Thermotest. Different adjacent substrates were used in this apparatus. Good agreement was obtained between tests, and the slight temperature differences between American, British and French methods have little significance. The A.A.T.C.C. Tentative Test Method 31-52 is suggested as the norm. S.R.C.

Dyeing Polyester Fibres with Aid of Carriers. Theoretical and Practical Considerations

P. Rochas *Teinture et Apprêts*, No. 58 (June 1960) 76-86

The maximum quantity of carrier taken up by polyester

fibres is the same for the majority of carriers, namely 70 mg. carrier per 1 g. of fibre. If the concentration of carrier in the bath is higher than that necessary to bring the concentration in the fibre to 70 mg./g., the amount of dye taken up decreases in many cases, the explanation being that any excess of carrier retains dye in the bath. A two-stage process is therefore suggested, i.e. treating the material in a solution of carrier, rinsing to remove excess, and dyeing in a separate bath. Carriers can be removed from the fibre completely by treatment with alkali, but even after such treatment the fibre retains ability to take up increased amounts of dye. It is concluded that the carrier has a permanent opening-up effect on the fibre structure. The swelling in water of the fibre is not affected by the carrier treatment. The carriers cause shrinkage of the polyester fibre. If this shrinkage is opposed, no increase of dye uptake is observed. W.M.

Dichroism of Dyes in Stretched PVA Sheet. III—Direction of Absorption of Pinacyanol Iodide

Y. Tanizaki

Bull. Chem. Soc. Japan, 33 (July 1960) 979-985

Dichroism of pinacyanol iodide in PVA (polyvinyl alcohol) sheet is recorded for wavelengths longer than 220 mμ, and at least 16 absorption bands are recognised. The relation of R_d (optical density ratio) to R_s (stretch ratio) of the sample sheet, when analysed concerning same absorption bands, afforded 2 groups of transitions, one existing along the long axis of the mol., the other being at right angles to it. The latter is shown to be due to the two orthogonal electronic transitions localised in the quinoline nuclei of the mol. The transitions along the long axis consist of 2 groups each involving several bands which are distributed with nearly constant distance of 1500 cm^{-1} between them. It is deduced that the mol. is not planar. H.H.H.

PATENTS

Aqueous Dispersions of Diazo Components and of their Mixtures with Coupling Components

Whiffen & Sons *BP* 840,357 (2 Oct 1957)

Dispersions having no tendency to oil when heated are obtained by dissolving a diazo component or mixture of a diazo component and a coupling component in a water-miscible solvent and then adding this solution to an aqueous solution of an anionic dispersing agent. Thus dianisidine (C.I. Azoic Diazo Component 48) is dissolved in methanol and the solution dispersed in an aqueous solution of Na oleyl *p*-anisidine sulphonate, Na acetate and acetic acid. Cotton cloth is treated in the resulting dispersion, and the dyeing diazotised and treated with phloroglucinol. C.O.C.

Oil-in-water Emulsions containing Methylcellulose for Pigment Dyeing and Printing

Interchemical Corp. *USP* 2,917,475 (24 July 1956)

Powdered methylcellulose is dispersed in a synthetic resin solution in a volatile water-immiscible organic solvent along with Na lauryl sulphate and an amine salt of a fatty acid to form a concentrate containing methylcellulose (10-25% by wt.), organic-solvent-soluble binder resin (5-22), amine salt of a fatty acid (1-5), volatile water-immiscible organic solvent or solvent and water (> 5) and Na lauryl sulphate (0.6-1.2 parts per part of methylcellulose). This concentrate is readily diluted with water and volatile water-immiscible solvent to yield an oil-in-water emulsion-printing vehicle containing methylcellulose dissolved in the aqueous phase. C.O.C.

Dyeing with Pigments, and Resin Binders therefor

Ciba *BP* 842,727 (Switzerland 1 Dec 1955)

Dyeings of good fastness to migration, and satisfactory to good fastness to washing are obtained by use of an aqueous bath containing a pigment dispersed with a cation-active dispersing agent and as sole binders a cation-active dispersion of an addition polymer free from anion-active water-solubilising groups and an aqueous dispersion, produced with a cationic dispersing agent of a hardenable aminoplast resin. This resin is insoluble in water but soluble in organic solvents. Both dispersions are of the oil-in-water type. After being dried the dyed material is baked. The resulting dyeings are usually of better fastness to wet-rubbing than those produced by use of either of the binding agents alone.

BP 842,728 (Switzerland 30 Nov 1955)

Only one of the above binders is used but the fastness properties of the dyeing are increased if the dyebath contains in addition a water-soluble hardenable aminoplast resin. C.O.C.

Dyeing and Printing Cellulosic Textiles

FBY BP 837,750 (Germany 15 July 1957)

Dyeings and prints of outstanding fastness to wet processing on cellulosic textiles are obtained by use of a dye free from sulphonic and carboxylic acid groups and containing at least one $-SO_2NH-R-O-A$ group (R = divalent hydrocarbon radical providing a straight chain of 2 or 3 C between the N and O atoms; A = radical of a polybasic inorganic acid or an organic derivative thereof) and then heating in presence of an acid-binding agent. Thus an aqueous paste containing the sulphuric ester of Ni phthalocyanine - 3 - tetrasulphohydroxyethylamide, NaOH, urea, Turkey Red oil and gum tragacanth is padded on to cotton cloth which is then immediately heated to 120°C. for 10 min., rinsed with hot water, and soaped at the boil for 20 min. This yields a level greenish blue dyeing of very good fastness to washing and good fastness to solvents and to light. C.O.C.

Padding with Esters of Leuco Vat Dyes

FH BP 837,000 (Germany 2 July 1955)

Difficulties arising from differences in the affinities of dyes for the fibre and in their variation with temperature are much reduced if 3-10 g./l. of polyvinylpyrrolidone is added to the dye liquor or, alternatively, if the cloth is first impregnated with a solution of polyvinylpyrrolidone. C.O.C.

Dyeing Synthetic Polymer Fibres with Disperse Dyes

Courtaulds BP 838,735 (19 Sept 1957)

Octylphenol and octylresorol are good carriers for the disperse dyeing of cellulose triacetate, Terylene and acrylonitrile polymers and copolymers. C.O.C.

Disperse Dyeing of Polyester Fibres

FH BP 839,130 (Germany 24 Dec 1956)

A mixture of an ester of benzoic acid with an aromatic alcohol, phenol, or naphthol and an ether containing at least one aromatic radical is an excellent carrier for use in disperse dyebaths, e.g. a mixture of benzyl benzoate (4 parts), dibenzyl ether (4) and condensate of 1 mol. dodecyl-phenol and 13 mol. ethylene oxide (2). C.O.C.

Stable Carrier Compositions for Use in Disperse Dyeing

Courtaulds BP 839,707 (19 Sept 1957)

Substituted phenol carriers do not readily disperse in water by use of standard methods. This difficulty is overcome by using a mixture of soap, a water-miscible alcohol of b.p. > 75°C., water and a cyclohexylphenol, a substituted cyclohexylphenol or an octyl-phenol or -resorol. The soap forms 40-80% by wt. of the mixture. Such a mixture gives a fine and even dispersion when stirred into hot water. C.O.C.

Disperse Dyeing and Printing of Polyesters

BASF BP 839,307 (Germany 28 Sept 1957)

Treating the polyesters with diphenyl sulphone or its substitution products before or during dyeing and printing yields much deeper dyeings of improved fastness. C.O.C.

Dyeing Polyolefin or Polyvinylidene Chloride Filaments with Monoazo Disperse Dyes

Vereingte Glanzstoff-Fabriken BP 838,687 (Germany 25 Sept 1957)

Monoazo dyes whose aromatic nucleus or nuclei contain one or more straight or branched aliphatic side chains of > 6 C, e.g. the dye *p*-octodecylaniline \rightarrow β -naphthol, are readily absorbed by polyolefins or polyvinylidene chloride filaments from hot aqueous dispersion in absence of solvent or oil. C.O.C.

Improving the Dyeing Properties of Polymers Containing Nitrile Groups

Gevaert Photo Producten BP 838,296 (22 May 1956)

Treating polymers containing nitrile groups with hydroxylamine in aqueous medium at pH 2-7 gives a whiter product than is obtained at pH > 7 and there is less consumption of hydroxylamine. Thus polyacrylonitrile fibres boiled for 2 hr. with aqueous hydroxylamine

sulphate dyed red with C.I. Acid Red 88, whereas untreated fibres dyed only a pale pink. C.O.C.

Preventing Corrosion of Molten-metal Baths used in Dyeing

BASF BP 838,691 (Germany 23 Nov 1957)

Adding acetylenic mono- or poly-hydric alcohols, e.g. propargyl alcohol, to the aqueous dyebath floating on the molten metal prevents corrosion. C.O.C.

Reducing Agents for Vat and Sulphur Dyes (III p. 634)

Why are Substantive Dyes Substantive? II—Behaviour of Dyes in Electrolyte-free Solutions (IV p. 635)

Synthesis and Properties of Acid Monoazo Dyes (7th Communication) (IV p. 636)

Fluorescent Brightening Agents (IV p. 642)

Aqueous Dispersions for Coating Fibrous Materials (IV p. 643)

Action of Tetrakis(hydroxymethyl)phosphonium Chloride on Wool (VI p. 645)

Wool Dyeing and Finishing in the West of England (VI p. 645)

Fluorescent Brightening Agents and Blueing Agents in Wool Processing (VII p. 646)

Effect of Dye Concentration on Light Fastness of Dyed Fibres (XIV p. 656)

IX—PRINTING

Chitin Fibres and Chitosan Printing

Bao Chi Ming

Faserforsch. und Textiltech., 11 (July 1960) 320-326

The preparation of chitin and chitosan and their spinning into fibres and application as pigment-binding agents in dyeing and printing of textiles are described. Half-hydrolysed chitin fibres have good ion-exchange properties and may be used to de-ionise organic compounds. Fabrics dyed or printed with chitosan as binding agent have good wash-fastness, low dirt acceptability and do not become brittle on ageing. W.R.M.

Development of Carrageenates and Application in Textile Printing—II

H. Schulzen Melliand Textilber., 41 (Aug 1960) 992-995

It is claimed that carrageenates can replace gum tragacanth as a thickening agent. Instructions for preparation of stock solutions are given, and different classes of dyes with which carrageenates can be used, either alone or in combination with other thickeners, are mentioned. Carrageenates, unlike alginates, are not sensitive to acids, are hardly affected by alkalis, and do not react with metals. W.M.

Drying in Screen Printing

Fr. Lewisch Melliand Textilber., 41 (Aug 1960) 996-997

The different methods of drying printed pieces are reviewed, and heating of the tables is considered the best solution. W.M.

PATENTS

Printing Screens

Haver & Brecker BP 838,905 (Germany 21 Aug 1956)

The wire gauze forming the screen has one or both of its surfaces smoothed, e.g. by calendaring, with simultaneous reduction in its thickness. This gives substantial saving in colour and in wear of the squeegee and also yields prints with clearer and sharper edges. C.O.C.

Pigment Resin Printing Paste

Sumitomo Chemical Co. BP 837,507 (Japan 5 Apr 1957)

An aqueous solution of polyvinyl acetal (from polyvinyl alcohol or alkyl alcohol-modified polyvinyl alcohol and 0.1-0.5 mol. equivalent of an unsatd. aldehyde per vinyl alcohol unit), mixed with a water-dispersible pigment paste, provides a printing paste which yields fast, deep, and clear prints and does not need a high curing temperature but only cylinder drying. C.O.C.

Diazotype Materials Sensitised with N-Hetero-p-aminobenzenediazonium Salts

General Aniline BP 840,108 (U.S.A. 25 July 1957)

Benzenediazonium salts containing a 1,3-oxazolidino or 1,3-oxazino group *para* to the diazo group used as sensitisers in diazotype compositions result in prints of excellent density, opacity, and brilliance. C.O.C.

Photographic Images Consisting of Phthalocyanine Pigments

DuP *USP* 2,915,392 (20 Apr 1955)
A suitable support is coated with an organic solvent solution of a phthalocyanine intermediate complex and oxalic or an α -hydroxycarboxylic acid or their alkali-metal or ammonium salts. On exposure through a negative to light and then treatment with either a solvent for the intermediate or an aqueous solution of a strong acid, a positive image of a metal phthalocyanine pigment is formed. Thus filter paper was impregnated with a chloroform solution of the Cu phthalocyanine propigment Cu phthalocyanine $(Cl)_n(OCH_3)_n$ (Example 4 of *USP* 2,662,896 (J.S.D.C., 71 (1955) 558)), dried, treated with oxalic acid, dissolved in the solution of the propigment and dried. The paper was then exposed to sunlight through a negative for 6 min., treated with HCl in aqueous methanol and then with 2% aqueous ammonia. The acid decomposed the propigment, leaving the blue Cu phthalocyanine positive print on the paper. Alternatively the unexposed propigment is removed by washing with acetone before the acid and neutralising treatments are given.

C.O.C.

Machine for Printing Carpets (I p. 633)

Machine for Applying Transfers Continuously to Woven and Knitted Fabrics (I p. 633)

Reducing Agents for Vat and Sulphur Dyes (III p. 634)

Water-dispersible Polyester-Aminoplast Condensates—Coatings, Binders, etc. (III p. 635)

2,2'-Alkylen-bis-(1,3-cyclohexanediones) as Diazo-type Coupling Components (IV p. 639)

Film Carbon Paper (V p. 644)

Incorporation of Ultraviolet Absorbers in Textiles (VIII p. 646)

Aqueous Dispersions of Diazo Components and of their Mixtures with Coupling Components (VIII p. 647)

Oil-in-water Emulsions containing Methylcellulose for Pigment Dyeing and Printing (VIII p. 647)

Dyeing and Printing Cellulosic Textiles (VIII p. 648)

Disperse Dyeing and Printing of Polyesters (VIII p. 648)

Coating Organic Material on Organic Polymer Substrates (X p. 650)

Heat-sensitive Copy Paper (XI p. 654)

X—SIZING AND FINISHING

Determination of Interfacial Forces Between Water-repellent-finished Surfaces and Water and their Relation to the Water-repellent Effect

K. Hintzmann *Melliand Textilber.*, 41 (Mar 1960) 341-344

To compare the efficiency of different water-repellent agents, discs of filter paper are treated and the force required to separate the treated material from the surface of water is measured with the aid of a torsion balance. Results obtained are highly reproducible. W.M.

Preparation and Rates of Hydrolysis of Perfluoro Esters of Cellulose

R. Benerito, R. J. Berni, and T. F. Fagley

Text. Research J., 30 (May 1960) 393-399

Treating cellulose with perfluoro-octanoyl or perfluorobutyl chloride in dimethylformamide in presence of a tertiary aromatic amine yielded esters of 0-02-0-58 degree of substitution. They had excellent oil- and good water-repellency, which was durable to dry-cleaning and aqueous launderings with a neutral detergent. C.O.C.

Reaction of Dimethylol Urea with Cotton

P. C. Mehta and R. D. Mehta

Text. Research J., 30 (July 1960) 524-532

Determination of N, N-CH₂OH and N-CH₂N in treated fabrics shows that no one structure can be assigned to the polymers formed with different catalysts, e.g. with tartaric acid and NH₄ sulphate it is highly cross-linked whereas with other catalysts it is essentially linear. Cross-linking between polymer and cellulose is not a necessary requirement, in the former case, for producing crease recovery. Structures are proposed which agree with the known chemistry of urea-formaldehyde resins and also offer reasonable explanation of the wide difference in the fastness to washing of the finishes obtained with different catalysts. Formaldehyde, liberated during drying and curing, reacts with the cellulose in presence of acid

catalysts, and a small but significant amount of formaldehyde is detected in the fabric after complete removal of the resin by acid hydrolysis. This formaldehyde, presumably present as methylene bridges, renders the acid-hydrolysed fabric insoluble in cuprammonium hydroxide. Use of this insolubility as a test for cross-linking in treated fabric has been examined, but preliminary results obtained indicate that the test may not be valid. C.O.C.

Reaction of Dimethylol Ethylene Urea with Cotton

P. C. Mehta and J. R. Mody

Text. Research J., 30 (July 1960) 532-539

When the title compound is applied to cotton in presence of acidic or latent acid catalysts, the polymer formed has DP 2-5, the higher value being obtained with the more acidic catalysts. There were no marked differences in crease recovery with any of these catalysts. Alkaline catalysts gave poor resin fixation and crease recovery, indicating little or no polymerisation and reaction with cellulose. In absence of catalyst a significant amount of fixed resin was obtained but no improvement in crease recovery. Alkaline hydrolysis of the treated fibres with 0.5 N-NaOH at 35°C. gives an estimate of the free methylol groups present and the content of these shows good relationship with the loss in strength caused by chlorine retention and scorching. C.O.C.

Effects of Cross-linkage in Crease-shedding Cotton Fabrics

J. G. Frick, B. A. K. Andrews, and J. D. Reid

Text. Research J., 30 (July 1960) 495-504

With dimethylol urea, dimethylol ethylene urea, or formaldehyde maximum crease-recovery angles are obtained with a substitution of 0.04-0.05 cross links per anhydroglucose unit. Differences in the retained tearing or breaking strength of fabrics treated with these agents to give the same degree of crease recovery are caused by the differing degrees of degradation of the cellulose occurring during the application of the agents. None of the differences in retained strength is attributable to either the size or the structure of the cross links. The much higher losses with formaldehyde are caused by the higher acidity of the catalyst. C.O.C.

Studies of Wrinkle-resistant Finishes for Cotton Textiles

I—Release of Formaldehyde Vapours on Storage of Wrinkle-resistant Cotton Fabrics

J. D. Reid, R. L. Arceneaux, R. M. Reinhardt, and J. A. Harris

Amer. Dyestuff Rep., 49 (11 July 1960) 490-495

Finishes obtained by use of five classes of N-methylol finishing agents with each of three types of catalysts have been studied. Some finishes produce more of a hazard in formaldehyde evolution than others and this is also true of some catalysts. Reapplication of catalyst to a washed, treated fabric showed that presence of catalyst greatly increased the evolution of fumes. The most important means of reducing the amount of formaldehyde evolved from finished fabrics is the washing after baking. An accelerated storage test is described. In samples of finished fabric which had not been washed after baking, the amount of formaldehyde evolved by keeping them in sealed jars for 16 hr. at 80°C. is a function of the acid stability of the finish, whereas with samples that have been washed it is related to the methylol content of the finish.

II—Effect of Storage on Properties of "Wash-and-Wear" Cottons

J. D. Reid, R. M. Reinhardt, R. M. H. Kullman, and R. L. Arceneaux

Ibid., 49 (25 July 1960) 527-531

Determination of the chlorine resistance, breaking and tearing strengths, and crease recovery of the above samples showed that washing after baking is a critical factor affecting the durability of desired properties on storage. Cloth treated with a dimethylol ethylene urea was particularly liable to lose its resistance to chlorine on being stored, and cloth treated with a pyrimidine derivative and not washed after baking rapidly lost its crease-recovery powers on storage. Cotton treated with a triazone or melamine derivative also showed increased susceptibility to chlorine damage when not washed before storage. C.O.C.

"Minimum-iron" Finish of Cotton Fabrics and the Problem of Chlorine Retention

W. Rümens, H. Götz, and R. Zeidler

Melliand Textilber., 41 (Mar 1960) 330-338

Nitrogen-containing resins retain chlorine by binding it to the N atom. In spite of this, N-containing resins of the reactant type can be successfully used. Especially good results are obtained with dimethylol-dihydroxyethylene urea. Conditions of condensation (catalyst, temp.) are of importance. Still better results are obtained by the simultaneous use of methyloltriazone. The latter compound—apart from its finishing effect—reacts strongly basic and neutralises any HCl liberated on heat treatment.

W.M.

History of the Anti-felting Finish

H. Stuessig *Melliand Textilber.*, 41 (Mar 1960) 323-325

The various theories to account for the felting tendency of wool are reviewed. The importance of the scale structure is stressed and it is then shown how the theoretical considerations influenced the different suggestions concerning anti-felting finishes.

W.M.

Chemical and Mechanical Examination of Shrink-proof-finished Knitwear

J. F. Diehl *Melliand Textilber.*, 41 (Mar 1960) 325-329

Samples of knitted wool were subjected to 19 different anti-felting treatments. With wet-chlorination, the anti-felting effect depended on the amount of active chlorine. With 4%, good resistance is obtained, but fibre damage occurs especially under acid conditions. Dry-chlorination gave good resistance, but somewhat harder handle; solubility indicates fibre damage; tensile strength and extension at break are, however, not affected. Oxidation processes gave unsatisfactory results. Alkali-alcohol method imparts moderate to good shrink resistance. Application of enzymes was unsuccessful.

W.M.

Application of U.V. Absorbers to Terylene

P. T. Strandring and G. W. Penmore

J. Textile Inst., 51 (Aug 1960) T 336-T 338

Degradation of Terylene by sunlight is associated with strong absorption in a very narrow band of the ultraviolet region. This contrasts strongly with the behaviour of polyamide fibres where degradation is accompanied by weaker absorption over a wider range of wavelengths. This explains some differences in the weathering properties of polyester and polyamide fibres. Application of (a) 4% of 2-hydroxy-4-methoxybenzophenone and (b) 3% of phenyl salicylate to bright Terylene (containing < 0.1% of TiO₂) led to four- and three-fold increases, respectively, in the life of the Terylene. They were applied in the dye-bath and by a pad-cure process; by each method they are fast to washing and prolonged soaking in cold water. In temperate zones cloud and industrial haze remove a large part of the 295-315 mμ radiation, but the use of these agents has merit for light-weight Terylene fabrics, fine twine or film, particularly if they are liable to be exposed to intense sunlight.

C.O.C.

Reaction between Polyvinyl Alcohol Fibre and NN'-Methylenebisacrylamide

H. Ito

J. Soc. Textile Cellulose Ind. Japan,

16 (Jan 1960) 21-24

The use of NN'-methylenebisacrylamide in cross-linking polyvinyl alcohol is studied by the curing method (110-205°C.) using NaOH as catalyst. Reactivity of the heat-treated polyvinyl alcohol is less than that of the untreated. The maximum degree of substitution is 2.5% per mole, but even amounts greater than 1.5% render the reaction products insoluble in boiling water. However, the shrink-resistance is insufficient even after formalisation, although affinities for a wide range of dyes are increased.

L.P.

Aromatic Compounds of Phosphorus

I. Reichel and D. Purdela

I—Phosphorous Esters of the Anilide and of the 2-Naphthalide of 2,3-Hydroxynaphthoic Acid

Studii și Cercetări, Științe Chimice (Timișoara), V (July-Dec 1958) 71-86

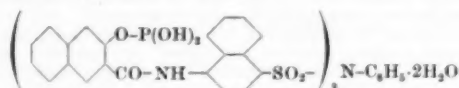
Condensation of 2,3-hydroxynaphthoic acid with 2,5-aminonaphthol-7-sulphonic acid in dimethylaniline and with an excess of PCl₅ gives mixtures of primary, secondary and tertiary phosphorous esters of 2,3-hydroxynaphthoic acid and of its arylide. These cpd. are separated and decomposed by alkaline hydrolysis into more simple

products. The rôle of dimethylaniline as medium is essential and consists in binding the liberated HCl, for if it is replaced by chlorobenzene only the anilide of 2,3-hydroxynaphthoic acid is formed, and production of phosphorous esters inhibited. Similar condensations occur in aniline and 2-naphthylamine. The esters may be applied to wool and cotton which have been previously mordanted with tannin and tartar emetic, and the treated textiles may be developed in solutions of diazonium cpd.

III—Action of excess PCl₅ on the reaction of Naphthionic Acid with 2,3-Hydroxynaphthoic Acid and upon their Acylated Compounds in a Medium of Dimethylaniline

Ibid., VI (Jan-June 1959) 95-100

A single substance is formed in each case at 120-140°C., which corresponds to the formula—

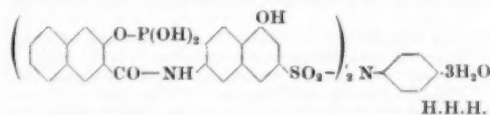


Similar reactions occur when the naphthionic acid is replaced by 1,5- or 1,8-naphthylamine sulphonic acid or by 1-aminonaphthalene-3,6,8-trisulphonic acid.

IV—Action of excess PCl₅ on the 2'-(5'-oxy-7'-sulpho)-Naphthalide of 2,3-Hydroxynaphthoic Acid in Dimethylaniline

Ibid., VI (July-Dec 1959) 95-102

A number of cpd. have been isolated according to the amount of PCl₅ used and the reaction procedure employed, the main one corresponding to the formula—



H.H.H.

Theoretical Considerations of the Stability of Dispersions of Solids in Organic Liquids

M. Bender

Off. Dig. Fed. Soc. Paint Technology,

32 (June 1960) 880-887

Factors involved in the stability of dispersions of solids in organic liquids that are of interest in the field of surface coating are: solvation, Brownian movement, attractive forces, stabilising agents, and electric charge. Importance of molecular polarity is stressed, and reference is made to the interaction of these factors as explanation of given rheological structures.

J.W.D.

Synthetic Polymers of Petroleum Origin for Coatings

D. F. Koenecke and W. L. van Nostrand

Off. Dig. Fed. Soc. Paint Technology,

32 (June 1960) 832-855

Copolymerisation of butadiene and styrene can be regulated to yield a polymer of comparatively low mol.wt. and with fairly high reactivity; this can be modified by the introduction of OH or COOH groups at active sites along the chains. Finishes based on the polymer, modified or unmodified, can be cured by air-drying, cross-linking, baking, or flame-curing. Resultant films are tough, hard, flexible, and of good chemical resistance, but residual reactivity and susceptibility to oxidation in ultraviolet radiation limit their weather resistance; in this they are inferior to phthalic alkyd and nitrocellulose varnishes. Flexibility and impact resistance can be controlled by the addition of reactive modifiers.

J.W.D.

PATENTS

Coating Organic Material on Organic Polymer Substrates

DuP

BP 839,483 (U.S.A. 5 June 1956)

The substrate is exposed in absence of oxygen to ionising charged particle radiation of energy 15-50,000 electron volts for a minimum exposure of 0.01 watt-sec./sq.cm. The organic coating, which must be dissimilar to the substrate, is applied while the effect of the irradiation is still active. The process is particularly useful in the sizing of organic polymer fibres; in the application of finishing agents, antistatic coatings, and colouring materials to fabrics; waterproof coating of regenerated cellulose film, etc.

C.O.C.

Wet Fixation of Aminoplasts on Fibres

Ciba BP 842,402 (Switzerland 8 May 1956)
Partial esters of glycerol with a monobasic acid or esters of glycol or a glycol monoether with a monobasic acid or an ester of a polybasic acid with a monohydric alcohol, e.g. diacetin, mono- or di-chlorohydrin, glycol diacetate, glycol ethyl ether acetate and diethyl tartrate, used as catalysts in aqueous aminoplast liquors used for impregnating textiles, enable the treated goods to be cured without being dried. Thus cotton bunks impregnated with a melamine-formaldehyde precondensate (1:2 mol.), formaldehyde and dichlorohydrin were heated for 1 hr. at 130°C. in an autoclave. This fixed all the resin taken up and none was removed by boiling for 30 min. in distilled water. C.O.C.

Flame-resistant Finish

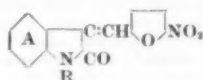
FH BP 842,393 (Germany 24 Dec 1955)
When impregnation with a chlorinated paraffin wax and a metal oxide, especially antimony trioxide, is used, addition of a chlorinated polyethylene and/or polypropylene or of a chlorinated ethylene-propylene copolymer, prevents the treated goods having a moist and sticky handle. It also increases the fastness of the finish to dry and wet rubbing. C.O.C.

Water-repellent Finish

Boehme Fettchemie BP 835,797 (Germany 15 June 1956)
Materials of all kinds are given an excellent water-repellent finish by impregnating or coating them with an organic solvent solution of the product obtained by treating a metal alcoholate with an acid high mol.wt. organic derivative of a phosphorus acid. Thus cotton cloth is treated with a solution in trichloroethylene of paraffin wax (m.p. 51°C.), acetic acid and the condensate of 1 mol. Al isopropylate and 0.8 mol. acid diethyl phosphate. On drying at room temperature or higher a water-repellent finish is obtained whose effectiveness depends on the amounts and the proportions of the agents used. C.O.C.

Indole Derivatives for Rendering Natural and Synthetic Polymers Self-sterilising and Bactericidal

ICI BP 836,477 (25 Oct 1957)
Compounds of formula—



(R = H or acyl; nucleus A may be substituted) when adsorbed by polymers still retain their bactericidal properties. Thus cellulose and its derivatives when entered into an aq. soln. of 3-(5'-nitro-2'-furfurylidene)-oxindole (1 in 200,000) at 76°C. and well agitated for 10 min. so that the solution cools to 30°C., and dried, have pronounced bactericidal property. C.O.C.

Silver Thiocyanate as a Bactericide

Permachem Corpn. BP 835,927 (12 Mar 1957)
Textiles, paper, leather, etc., are rendered bactericidal by impregnating them with silver thiocyanate, preferably by precipitating it *in situ*. C.O.C.

Bactericidal Synthetic and Proteinaceous Fibres by Use of Neomycin

American Cyanamid Co. BP 842,217 (U.S.A. 7 Feb 1957)
Neomycin is substantive to the above fibres to which it imparts a durable bactericidal finish, e.g. wool and nylon padded with their own weight of 0.1% aq. neomycin and dried for 2 min. at 225°C. were given an excellent bactericidal finish which was unimpaired by commercial dry cleaning. C.O.C.

Anti-static Finish

FH BP 839,456 (Germany 4 June 1955)
Use of an aqueous solution of a salt of an $\alpha\beta$ -unsaturated dicarboxylic acid and a vinyl compound in presence of a water-soluble polyglycol or a copolymer of ethylene oxide and other ethylene oxides of mol.wt. 400–10,000 or a polycondensate containing > 1 free OH group of polyglycols and bivalent acylating agents and drying at > 100°C., imparts an anti-static finish of good fastness to washing. C.O.C.

Improving Shaped Products Containing Carbon-amide Groups. Anti-static Finish

Boehme Fettchemie BP 839,891 (Germany 16 Aug 1956)
Nylon, wool, or other shaped material containing carbon-amide groups is treated with aliphatic lactones or lactams or aliphatic or aromatic sultones in presence of a basic substance so that the weight of the material is increased by > 3%. There is no change in physical properties and the mechanical properties and colour are almost unaltered. The increase in weight is fast to washing. Thus nylon 6 staple fibre is saturated with 20% aq. NaOH, left for 30 min. and then treated in aqueous 30% solution of propiolactone. The exothermic reaction is first moderated by cooling and then allowed to proceed at 30°C. for 90 min. The treated fibre shows little increase in weight and is light coloured and hydrophilic. It has an excellent anti-static finish which is fast to washing, and good affinity for basic dyes. C.O.C.

Imparting Dimensional Stability to Cellulosic Fabrics

Tootal Broadhurst Lee Co. BP 838,394 (23 Mar 1957)
The fabrics are treated with formaldehyde or other aliphatic aldehyde dissolved in an ionising solvent. The solution also contains an acid salt of a polybasic acid, having a first dissociation constant in water of < 10⁻⁴, with a metal of which a less-acid salt with the same acid is precipitated before the acid salt on removal of solvent from and/or on the solution being heated. Thus spun viscose rayon cloth was impregnated with 80% of its weight of a solution of Pb nitrate (110 g.) in water (1000 c.c.) and dried at 60°C. It was then further impregnated with 80% of its weight of a solution of potassium hydrogen phthalate (68 g.), water (250 c.c.) and 40% formalin (750 c.c.) previously neutralised with NaOH, and dried at 60°C. The finished fabric contained 0.06% fixed formaldehyde, had excellent resistance to abrasion and excellent dimensional stability to laundering. C.O.C.

Permanent Crease-recovery Finish on Cellulose Textiles

Traitements Chimiques des Textiles BP 837,508 (France 11 Apr 1957)
The material is impregnated with natural or synthetic rubber latex, dried, treated with a monochloromethyl or polychloromethyl ether, dried and then baked. The treated material does not need ironing after laundering, does not retain chlorine, and has excellent dimensional stability even when repeatedly washed. Thus cotton cloth is impregnated with a 2% (solids content) aqueous suspension of latex, dried, impregnated with pyridinium salt of ethanediol-1,2-dichloromethyl ether (60), Na acetate (20) and water (1000), dried at 50–60°C., baked for 10 min. at 145°C., washed, and dried. C.O.C.

Flame-resistant Finish for Cellulose Textiles

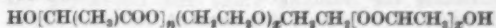
Courtaulds BP 835,581 (31 Dec 1956)
The textiles are impregnated with an aqueous solution of an aminoplast resin precondensate and a compound of formula—



e.g. 2-chloroethylaminophosphonate, dried and baked. C.O.C.

Polyglycol-Polyacid Ester Treatment of Textiles

American Cyanamid Co. USP 2,917,410 (20 June 1955)
Esters or ether-esters of mol.wt. 400–225,000 containing a polyoxyalkylene chain of mol.wt. 200–25,000 with a polyacid residue chain or chains of mol.wt. 200–200,000 attached at one end or ends of the polyoxyalkylene chain, are useful for imparting improved strength and resistance to wear of textiles. Thus viscose rayon challis impregnated with 2.5% of various diesters of approximate formula—



(n = 1–54; x = 63–74) had increased tensile strength and a large increase in Elmendorf Tear Strength, in some cases the strength being almost double that of the untreated control. C.O.C.

Imparting Permanent Pleats and Soil- and Abrasion-resistance to Cellulosic Textiles

U.S. Sec. of Agriculture USP 2,917,412 (12 May 1958)
The textiles are impregnated with an aqueous solution containing dimethylol cyclic ethylene urea (1–15% by wt. of solids content), polyethylene (0.1–5.0) and a latent catalyst

(0.1–5.0), e.g. Zn nitrate or 2-amino-2-methyl-1-propanol hydrochloride. They are then dried, preferably at < 45°C. and cured, e.g. on a tenter, calendered and pressed, at 120–220°C. The pleats resist at least 20 launderings and the garments do not require ironing even after being centrifuged. C.O.C.

Rayon Plissé Fabrics

American Viscose Corp.

BP 837,010 (U.S.A. 23 Dec 1955)

The fabric is impregnated with an aqueous solution at pH 1.2–2.5 of a cellulose ether, an aldehyde or aldehyde-alkylene glycol condensate and a catalyst, cured, scoured and then printed with NaOH to impart a plissé effect. Thus rayon shirting was impregnated on a pad with 75–85% of its weight of aqueous solution of hydroxyethylcellulose (5%), 37% formalin (10), mixture of sorbitan monopalmitate and tristearate (0.5), NaHSO₄ (2) and Na₂SO₄ (0.5). Padding was carried out at a tension giving 3–8% stretch. It was then tenter-dried at 115–140°C., baked for 150 sec. at 162°C., scoured at pH 9.0–9.5 for 15 min. at 50–75°C. Finally it was dried, wetted to 25% moisture content, printed with 50% aq. NaOH thickened with starch ether, and after a few minutes neutralised with acetic acid, washed, dried in absence of tension and steam-framed. The finished fabric had a plissé pattern. C.O.C.

Even Chlorination of Textiles Containing Wool

CFM

BP 838,036 (Germany 21 May 1957)

Even chlorination is obtained in an acidic aqueous bath if the condensate of a free polyalkylene polyamine and an alkylating agent is present. Both components of the condensate must be free from hydroxyl groups and the alkylating agent must contain more than one alkylating group. The preparation of these condensates is described in GP 874,757. C.O.C.

Reducing Felting Power of Wool Fabrics

Rohm & Haas

USP 2,915,419 (21 Nov 1956)

The fabric is impregnated with an aqueous dispersion of a polyester-aminoplast condensate and then heated to > 212°F. 26 examples of suitable condensates are given. C.O.C.

Permanent Setting of Wool Goods

Commonwealth Scientific and Industrial Research Organisation

BP 839,516 (Australia 2 July 1957)

The goods are heated at < 212°F. in presence of an aqueous solution of NH₄ thioglycollate. Thus pleated wool cloth was pressed for 5 sec. in a Hoffman press. The press head was then raised and the edges of the pleats were sprayed with an aqueous solution containing (by volume) 2% thioglycolic acid, 2% 0-880 ammonia and 0.1% wetting agent. It was then re-pressed with steam for 20 sec., the steam shut off and the cloth left in the press for a further 20 sec., followed by vacuum in the usual manner. The cloth was then dry-cleaned and without re-pressing immersed in cold water and then agitated in a detergent solution at 50°C. for 30 min., rinsed and air-dried, when it retained very sharp pleats. Control cloth similarly treated without being sprayed lost its pleats on immersion in the cold water. C.O.C.

Removing Discoloration Caused by Sulphur in Viscose Rayon

Lustrafil

BP 837,647 (28 Oct 1957)

Treatment with a sequestering agent first in acid and then in alkaline medium completely removes all coloured impurities from undersulphurised or incompletely desulphurised viscose rayon, e.g. treated with ethylenediaminetetra-acetic acid first at pH 4.0–4.5 and then in a second bath at pH 8.5–10.5. C.O.C.

Reducing the Swelling Value and Improving the Stability of Crimp of Regenerated Cellulose Fibres

Spinnfaser

BP 841,401 (Germany 29 Jan 1957)

Treatment for a few minutes with liquid ammonia at –60° to –35°C. and then rinsing with cold water results in the swelling value of the fibres being reduced and in improved stability of crimp, wet tenacity and loop strength. Thus spun viscose rayon costume cloth was treated for 6–10 min. in liquid ammonia at –60°C., rinsed with cold water while under tension on a stenter and dried. This reduced the swelling value of the rayon by about 30% and considerably improved the stability of its crimp. C.O.C.

Producing a Pile on Flat Tricot Knitted Fabric

Dow Corning Corp.

USP 2,914,836 (25 June 1957)

A flat tricot knitted fabric, in which a large proportion of the staple fibre is a synthetic organic material, is treated with a methylsilicone fluid, raised, passed near an electrostatic bar to cause the pile to stand out, again raised and finished in the usual manner. This produces an excellent pile far superior to that obtained if the silicone is applied after the first raising or after the passage close to the electrostatic bar. C.O.C.

Reducing the Tendency of Spun Polyethylene Terephthalate Fibres to Pill

ICI

BP 840,796 (31 May 1955)

Reducing the intrinsic viscosity of the fibres to 0.45–0.28 at any stage of their manufacture or processing results in fabrics containing them having less tendency to pill. Thus spun polyethylene terephthalate fabric is treated with 0-880 ammonia for 18–66 hr. in a sealed aspirator at room temperature and pressure. The resistance to pilling varies with the length of treatment, there being no pilling after 66 hr. treatment. The intrinsic viscosity varies from 0.59 for the untreated to 0.38 for fibres treated for 60 hr. and the resistance to abrasion also deteriorates as the time of treatment is lengthened. C.O.C.

Treating Polymers Containing Nitrile Groups with Hydroxylamine

Gevaert Photo Produkten

BP 786,960 (24 Sept 1954)

BP 840,797 (2 June 1955)

Treatment of nitrile-containing polymers with hydroxylamine much enhances their affinity for acid dyes. The treatment may be given before or after the polymers have been formed into fibres. C.O.C.

Modifying Synthetic Condensation Polymers

DuP

BP 838,412 (U.S.A. 6 Apr 1955)

The polymers are subjected to ionising radiation while in intimate contact with a modifier consisting of at least one organic compound which when chemically bonded with the polymer as a result of the irradiation causes appreciable change in the polymer properties. Thus polyester fabric coated with liquid bis(3-aminopropoxy)ethane and then irradiated to a total dosage of 40 Mrp, given 15 washings and then dyed with C.I. Acid Red 134 from an ammonium acetate bath is bright red, whereas an uncoated irradiated sample and a sample of the original fabric similarly treated remain undyed. C.O.C.

Mineral Fibres Coated with Synthetic Resins

Owens-Corning Fiberglass Corp.

BP 842,634 (U.S.A. 27 Dec 1955)

Glass and mineral wool fibres are given tough, hard resistant coatings of improved flexural and compressive strength by treating them with an aqueous dispersion of a thermosetting phenol-aldehyde or aminoplast resin and then baking. To prevent the resin being degraded by alkali generated at the fibre surface or present in the coating composition, there is added to the composition 0.25–1.0% (on the total dry solids of the composition) of a salt of a metal whose hydroxide, silicate and carbonate in aqueous solution have at any strength pH > 10.5, e.g. Al sulphate. C.O.C.

Leather Substitute

DuP

BP 842,492 (U.S.A. 14 Jan 1957)

A coating composition which can be applied to a wide variety of substrates to yield products which look and feel like leather comprises a polymer or copolymer of vinyl chloride, a plasticiser and 5–40% on the weight of the binder of dyeable organic fibres > 3000 μ. long and of refractive index differing from that of the binder by < 0.1. C.O.C.

Permanently Coated Carrier Webs

Dornbusch & Co.

BP 838,713 (Germany 23 Apr 1956)

A net-like or lattice plastic foil is formed in a mould roll having recesses engraved in it. The inner surface of the foil is then heated until it softens and the outer surface until it fuses, whereon a carrier web of fabric, paper or other fibrous material is pressed into the recesses of the roll so as to be firmly secured to the plastic foil. The products are highly resistant to wear, porous and pervious to air and water vapour. C.O.C.

unaltered and does not impair the mechanical properties of the fibre.

W.M.

Relation between Thickness and Opacity of Paper

R. Sève

Assoc. tech. ind. papetière, Bull. No. 5 (1959) 256-264
Chem. Abs., 54 (25 June 1960) 12580

Variation in opacity (measured by the contrast ratio and printing opacity methods) have been studied as a function of paper thickness, a wide range of weights of paper and various layers of superposed sheets being used. Using the Kubelka-Munk formula, the theoretical relation between contrast ratio, opacity, printing opacity, and thickness was obtained and confirmed experimentally. Tables and graphs for converting TAPPI printing and contrast opacities to AFNOR (French standard) contrast opacity are given.

C.O.C.

PATENTS

High-gloss Coating of Fibrous Sheets

M. F. Conant BP 842,879 (U.S.A. 26 Sept 1955)

Both sides of the sheet are coated with a solution of a plastic in a volatile solvent, subjected to radiant heat to evaporate off the solvent, and then cooled. During the heating and cooling the sheets are carried by an endless loop conveyor having upwardly directed sharp-pointed pins on which the sheets rest. The pins are carried on crossbars which are far apart to permit the radiant heat to pass through the upper run of the conveyor to heat the lower sides of the sheets.

C.O.C.

Heat-sensitive Copy Paper

Minnesota Mining & Mfg. Co.

USP 2,916,395 (21 July 1958)

Aqueous Dispersions for Coating Fibrous Materials (IV p. 643)

Film Carbon Paper (V p. 644)

Unit Cell of Cellulose Triacetate (Variety I) (VI p. 645)

Coating Organic Material on Organic Polymer Substrates (X p. 650)

Leather Substitute (X p. 652)

Coating with Powders (X p. 653)

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Moiré Effects on Furs

S. Beyer

Kožářství, 9 (1959) 364

J. Amer. Leather Chem. Assoc., 55 (Aug 1960) 470

The cold-wave methods employed by hairdressers are not effective. Solutions containing Na_2SO_3 , mercapto compounds, thiourea, fatty alcohol sulphonates, non-ionic detergents and protein condensates are brushed on the furs, which are then embossed and sprayed with 6% aq. H_2O_2 , followed by lactic acid. After being dried, the furs are dyed as usual. The moiré is durable, but dyeing is usually not entirely level.

C.O.C.

Theory and Practice of Leather Finishing by Pigment Colours in the U.S.S.R.

V. I. Eliaseva

Kožářství, 9 (1959) 102-108

Chem. Abs., 54 (25 June 1960) 12625

A study of the behaviour of pigment-finished leathers by the wearing of experimental shoes. Instruments for determining flexing resistance, abrasion, heat stability and mechanical properties are described. Resin dispersions penetrate better in leather the smaller their particles, the greater their resistance to salting-out and the lower the surface tension. The most important factor is the potential difference between the dispersed particles and the leather fibres. A dispersion of negatively charged particles should have pH > isoelectric point of leather if maximum penetration is to be obtained. Examples of formulation of the pigment colours based on these principles are given.

C.O.C.

Aqueous Dispersions for Coating Fibrous Materials (IV p. 643)

Uneven Dyeing of Sheep and Lamb Fur Skins Fixed by Formaldehyde (VIII p. 647)

Use of Pyrocatechol (C.I. Oxidation Base 26) in Fur Dyeing (VIII p. 647)

Even Dyeing of Furs (VIII p. 647)

Coating Organic Material on Organic Polymer Substrates (X p. 650)

Coating with Powders (X p. 653)

XIII—RUBBER; RESINS; PLASTICS

New Developments in the Soluble Vinyl Aromatic-Diene Copolymer Paint Resin Field

D. A. Hilliard

Off. Dig. Fed. Soc. Paint Technology.

32 (June 1960) 856-879

Properties of a soluble vinyl-toluene-butadiene copolymer resin, Pliolite VT (Goodyear), are described with special reference to its solubility, compatibility, drying rate, chemical resistance, oxidation (which improves the durability of the resultant film), and effect of ultraviolet radiation. The performance of traffic paints, floor enamels, multicolour paints, and metal primers based upon the resin is described. There are 33 photographs.

J.W.D.

Alkylated Phenols for Synthetic Resins Soluble in Oil. I—Alkylation of Phenol by Butan-1-ol

I. Nanu and I. Manovich

Studii și Cercetări, Științe Chimice

(Timișoara), V (Jan-June 1958) 145-160

Three sets of syntheses are described in which phenol and butan-1-ol were treated, respectively, in the presence of anhyd. H_3PO_4 , ZnCl_2 under reflux, and ZnCl_2 accompanied by continuous dehydration. The third process gave the best results. Fractional distillation of the crude alkylated phenol formed removed the small amounts present of unchanged phenol and of some tributylphenol, and afforded a fraction rich in isomeric monobutylphenols, which, after elimination of a small quantity of phenol ethers, was used for the synthesis of resins sol. in oil. There are 41 references.

H.H.H.

Some Phenolic Resins Soluble in Oil. I—Butyl Phenol-Formaldehyde Resins

I. Nanu and I. Manovich

Studii și Cercetări, Științe Chimice

(Timișoara), V (July-Dec 1958) 147-160

Methods are described for obtaining useful hard resins sol. in drying oils from the fraction of isomeric monobutylphenols reported in the previous abstract. The resins are very complicated polymol. mixtures, and the effect of the dibutyl deriv. present is found to be a stabilisation of linear development by the contribution of end groups. There are 28 references.

H.H.H.

PATENTS

Protecting Polyethylene Film Against Degradation by Ultraviolet Radiation

DuP

USP 2,917,402 (23 July 1958)

At least one surface of the film is coated with a volatile organic solvent solution of (1) a copolymer of 85-15% of vinyl fluoride and 15-85% of a vinyl ester of an aliphatic monocarboxylic acid of 2-20 C and (2) 5-25% (on total solids) of an ultraviolet absorber. Thus a copolymer of vinyl acetate and vinyl fluoride (15.6 and 84.4%, respectively) mixed with dibenzoyl resorcinol (16% by weight of solids) applied as a 0.04 mil. coating to polyethylene film, took 1500 hr. to total failure under an accelerated weakening test, whereas the untreated film failed in 350 hr.

C.O.C.

Pigmented Vinyl Chloride Polymers

Western Electric Co. BP 842,587 (U.S.A. 30 Jan 1957)

The polymer granules are heated below their m.p. with wettable dry powdered pigment and a liquid plasticiser which will wet the pigment and be partly absorbed by it. Treatment continues until the plasticiser is absorbed by both the pigment and the polymer so that the pigment particles are dispersed upon and adhere firmly to the granules. The amount of plasticiser is such that the final pigmented composition is dry and free flowing. The products have high chromaticity and, when used to colour resinous masses, a high proportion of the strength of the pigment present is utilised.

C.O.C.

Polymerisation within Wool Fibres (VI p. 645)

Intrinsic Viscosity and Molecular Weight of Polyethylene

Terephthalate (VI p. 645)

Pigmented Polyesters (VI p. 646)

Coating Organic Material on Organic Polymer Substrates (X p. 650)

- Improving Shaped Products Containing Carbonamide Groups. Anti-static Finish (X p. 651)
 Treating Polymers Containing Nitrile Groups with Hydroxylamine (X p. 652)
 Modifying Synthetic Condensation Polymers (X p. 652)
 Permanently Coated Carrier Webs (X p. 652)
 Coating with Powders (X p. 653)

XIV—ANALYSIS; TESTING; APPARATUS

Indicator for the Titration of Calcium Plus Magnesium with (Ethylenedinitrilo)tetraacetate

F. Lindstrom and H. Diehl

Anal. Chem., **32** (Aug 1960) 1123–1127

A new $\alpha\omega$ -hydroxyazo indicator, 1-(1-hydroxy-4-methyl-2-phenylazo)-2-naphthol-4-sulphonic acid, is recommended for the titration of calcium plus magnesium with EDTA. It has the same colour change as Eriochrome Black T, but the colour change is clearer and sharper, and aqueous solutions of the indicator are stable indefinitely. It may be substituted for the older indicator without change in the procedure. P.B.S.

Characterisation of the Colour Quality of Indicator Transition

C. N. Reilley, H. A. Flaschka, S. Laurent, and B. Laurent

Anal. Chem., **32** (Sept 1960) 1218–1232

The quality of an indicator end-point is composed of the sharpness, dependent upon the various equilibria involved, and the colour quality. The latter involves the subjective perception of colour and is readily handled by combining the principles of tristimulus colorimetry with Beer's law. This leads to an equation which relates numerically the various colour parameters and which permits calculation of the relative quantities of inert dyes necessary for screening an indicator. P.B.S.

Chemical Applications of Complementary Tristimulus Colorimetry

C. N. Reilley and E. M. Smith

Anal. Chem., **32** (Sept 1960) 1233–1240

Complementary tristimulus colorimetry is applied to some chemical problems, i.e. colorimetric analysis of multicomponent mixtures, calculation of the pK 's of acids, and the formulae and stability constants of complex ions. As an aid in computation, details of an inexpensive analog computer are given. P.B.S.

Mechanised Conversion of Colorimetric Data to Munsell Renotations

W. C. Rheinboldt and J. P. Menard

J. Opt. Soc. Amer., **50** (Aug 1960) 802–807

A programme for a high-speed digital electronic computer is described for computing the Munsell renotations H, V, C corresponding to given CIE chromaticity coordinates x, y and daylight reflectance Y . This is equivalent mathematically to a three-dimensional co-ordinate transformation where two of the three transformation functions are given only numerically for a grid of discrete points. As this grid consisted of about 5000 points unevenly spaced, the major problem was to devise an economic scanning routine in order to find the point used in the interpolation. This was done by consistent use of vector algebra and with the help of an interpretive routine for vector operations. C.O.C.

New Method for the Detection and Rapid Gravimetric Estimation of Pyrimidone in Presence of its Principal Impurities

I. Dick

Studii și Cercetări, Științe Chimice (Timișoara), **V** (Jan–June 1958) 101–106

The semi-micro procedure adopted is based on the total and unique pptn. of nickel pyrimidone sulphocyanammin, $[\text{NiC}_5\text{H}_7\text{ON}_3\text{S}_2(\text{SCN})_2]$, from its soln. by treatment with NH_4SCN and $\text{Ni}(\text{NO}_3)_2$; the ppt. is weighed directly. All accompanying substances are not pptd. H.H.H.

Paper Chromatography of Synthetic Dyes

J. C. Brown

J.S.D.C., **76** (Sept 1960) 536–542

Photoreduction and Photo-oxidation Sensitised by Dyes

G. Oster

J. chim. phys., **55** (1958) 899–904

Chem. Abs., **54** (25 June 1960) 11657

Review, 39 references.

C.O.C.

Polarographic Analysis of a Mixture of Anthraquinones

V. E. Ditsent

Zavodskaya Lab., **24** (1958) 951–952

Chem. Abs., **54** (25 June 1960) 11803

The half-wave potential values in LiCl were -0.75 , -0.55 and -0.40 v. for 2-ethylantraquinone, tetrahydro-2-ethylantraquinone and octahydro-2-ethylantraquinone, respectively. These clearly defined waves were obtained for a mixture of the three in 80% methanol in 0.5 \times LiCl. Direct proportionality was observed between the height of the wave and the concentration of each of the cpd. determined in presence of the other two. Dilute 2 ml. of the solution with methanol in a 50 ml. volumetric flask; transfer 5 ml. of the diluted sample to a 50 ml. flask, add 20–25 ml. methanol, 10 ml. 2.5 \times LiCl and dilute with methanol. Place 10–15 ml. of the treated sample in a polarographic cell, pass H_2 through the solution for 30 min. and take the polarograms. C.O.C.

Factors affecting the Intensity of J-bands in the Absorption Spectrum of Water-Gelatin Solutions of Dibenzothiacarbocyanine Dyes

A. V. Borin, P. N. Zaleznyak, and I. A. Pobedonostseva

Zhur. Nauch. i Priklad. Fot. i Kinematografi.

4 (1959) 401–410

Chem. Abs., **54** (25 June 1960) 11778

Tables and graphs are given showing the maximum intensity of the J-band (I_{max}) in aqueous solutions of 3,3'-dimethyl-9-ethyl-4,5,4',5'-dibenzothiacarbocyanine chloride (4×10^{-3} M.) containing 0.1 and 5.0–10.0% of gelatin. Chloride concentration in the gelatin had little or no effect on I . In most gelatins I was greatest and least for gelatin concentrations of 0.01 and 1.0%, respectively. The J-band was much stronger where the gelatins were free from disaggregated fractions. Increase in I with gelatin concentration when the latter is $> 1\%$ is caused by increase in the concentration of carboxyl groups resulting from interaction of these groups with basic side chains. C.O.C.

Analysis of the Accuracy of Pycnometric Estimation of the Density of Pigments and Other Powders

K. Řeháček

Chem. průmysl, **9** (1959) 292–296

Chem. Abs., **54** (25 June 1960) 11619

In gravimetric analysis the relative average error of pigment density $\sigma_p(\rho_p)$ can be calculated from the average error of filling $\sigma(b)$ and from the volume of pigment V_p (in c.c.) by use of the equation $\sigma_p(\rho_p) = 141 \sigma(b)/V_p q_p$ (q_p = density of the pycnometric liquid). Similarly, in volumetric analysis $\sigma_p(\rho_p) = 141 \sigma(c)/V_p$ ($\sigma(c)$ = average error in volume measurement). In both cases change in temperature Δt causes $\sigma_p(\rho_p) = -100 V_p \beta \Delta t / V_p$ (V_p = volume of the pycnometric liquid and β = its coefficient of volume expansion). For pycnometers with a capillary stopper $\sigma(b)$ is approx. 6×10^{-4} for petroleum; for pycnometers having a circular mark line, $\sigma(c)$ can be calculated from the diameter of the neck (d) and the average error of the position of the meniscus $\sigma(h)$ according to the equation $\sigma(c) = \pi d^2 \sigma(h) / 4 = 4.7 \times 10^{-3} d^2$. The dependence of $\sigma_p(\rho_p)$ on $\sigma(b)$ and on $\sigma(c)$ for weights corresponding to 0.5, 1.0, 2.0 and 5.0 c.c. of pigment is given diagrammatically; the diagram can also be used to calculate $\sigma_p(\rho_p)$ that arises from difference in temperatures. Suggestions are made for increasing the accuracy of the standard methods of Czechoslovakia, DIN, ASTM, and others. C.O.C.

Centrifugally Accelerated Paper Chromatography of Chloroplast Pigments

J. M. Anderson

J. Chromatography, **4** (Aug 1960) 93–98

Chlorophyll a and b (C.I. Natural Green 3), violaxanthin (C.I. Natural Yellow 27), carotene (C.I. Natural Yellow 26), and lutein pigments were resolved from algal extracts using Whatman Nos. 1, 3 and 17 mm circles and accelerating the chromatograms by centrifuge. Chromatofuge R_f values were obtained at increasing r.p.m. and found to reach a max. and then decrease. Max. R_f values were compared and found similar to results by conventional methods. Advantages were narrow compact zones and a rapid (10–25 min.) development time, thus allowing separation and recovery of more labile chlorophylls and carotenoids. F.J.

Methods for Estimation of Fluorescent Compounds

A. Kling and J. Kurz

Melliand Textilber., 41 (Mar 1960) 339-341
Spectrophotometric measurement of the concentration of fluorescent compounds in solution and on textile substrates is discussed and methods for investigating the dependence of intensity of fluorescence on the wavelength of the exciting UV-radiation are described. W.M.

Determination of Lead in Paints at the 1% Level

M. W. Westgate, R. B. Shurts, and E. F. Adams

Natl. Paint, Varnish Lacquer Assoc., Sci. Sect., Circ. No. 773 (1956) 8-17
Anal. Abstr., 4 (1957) Abstr. 859
Chem. Abs., 54 (25 June 1960) 12611

Report of co-operative tests on determination of Pb in paints tinted with known amounts of Pb chromate (C.I. Pigment Yellow 34) (as liquids and dried films) by a standard method involving wet-ashing with HNO_3 - H_2SO_4 , isolation of Pb as PbSO_4 , and precipitation as PbCrO_4 . An improved procedure using Caro's acid for the wet-ashing has been developed. After the PbSO_4 produced has been extracted with aq. NH_3 acetate, a preliminary volumetric test on an aliquot of the solution to ascertain if the Pb content is likely to be $> 1\%$ is advantageous. A known amount of standard NH_4 molybdate solution is added in the tannic acid as indicator; a yellowish brown shows that the end-point has been exceeded and that the Pb is $< 1\%$. If $> 1\%$ Pb is indicated, the amount is accurately determined on the remainder of the solution by precipitation as PbCrO_4 . C.O.C.

Comprehensive Index of Methods of Test for Paints and Paint Materials

Off. Dig. Fed. Soc. Paint Technology, 32 (Aug 1960, Pt. 2) 1-64

An index, prepared by the Federation's Standard and Methods of Test Committee, of test methods found in (a) Federation approved methods, (b) A.S.T.M. Special Paint Compilation, (c) Federal Standard 141, (d) other Federal paint specifications, and (e) Canadian Govt. Specification Board Methods of Testing Paint and Pigments (1-GP-71A). Indexing is alphabetical and there is considerable cross- and multiple-indexing; specification titles are re-worded to bring together in the index different tests for the same property. M.T.

Fibre Analysis—Binary Mixtures of Secondary Cellulose Acetate with Other Fibres

Bull. Inst. Text. France (87) (Mar-Apr 1960) 107-110

A standard method is described which is based on preferential solution in acetone, for the analysis of mixtures of secondary cellulose acetate with fibres of wool, silk, regenerated proteins, cotton, regenerated cellulose, polyamides, polyesters, certain acrylics, and glass. J.C.F.

Light Scattering of Nylon 6.6 Dissolved in Formic Acid

P. R. Saunders *J. Polymer Sci.*, 43 (Mar 1960) 273-275

X-ray Studies on Polyvinyl Alcohol Fibre
H. Kawakami, N. Mori, H. Satō, and A. Miyoshi

I—Crystallinity and Orientation

J. Soc. Textile Cellulose Ind. Japan, 16 (Mar 1960) 155-160

Degree of crystallinity and orientation of stretched and heat-treated polyvinyl alcohol are determined by X-ray counter methods. The tensile properties and dye adsorption are also measured. The temperature, rather than the time, of heat treatment affects the crystallinity, and the latter increases with draw ratio. At draw ratio 1, the degree of orientation is nearly 0° , but increases abruptly to 84° at draw ratio 1.5, above which little further change occurs.

II—Crystallinity and Swelling of Heat-stretched and Relaxed Fibres

Ibid., 160-162
Swelling in water does not always depend solely on the crystallinity. The small-angle scattering in the meridional direction of the samples is measured, and the relation between crystallinity and swelling discussed by analysis of the X-ray data. L.P.

Determination of the Continued-tear Resistance of Fabrics

Textil-Rund., 15 (June 1960) 308-311
Standard method SNV 98 4 82 of the Swiss Standardisation Association. P.B.S.

Development of a Mechanical Test Equivalent to Five Average Hand Washings

R. B. Smith *Amer. Dyestuff Rep.*, 49 (25 July 1960) 533
Brief account of work done by the AATCC Committee on Colorfastness to Washing, RA60. C.O.C.

Study of the AATCC Accelerator Operating at Constant Power

H. P. Briar *Amer. Dyestuff Rep.*, 49 (8 Aug 1960) 559-564
Metering the amount of power applied to the test sample results in complex fabric parameter effects being minimised or eliminated from the results. This makes interpretation of the results much simpler. When comparing tests on the same fabric, the constant-speed method gives results that are independent of any fluctuations in the sample weight. C.O.C.

Colorfastness of Textiles to Commercial Laundering and Domestic Washing. Accelerated Tests. Tests No. IA, IIA, IIIA, and IVA. Tentative Test Method 61-1960

AATCC Committee on Colorfastness to Washing, RA60
Amer. Dyestuff Rep., 49 (25 July 1960) 534-535

Effect of Dye Concentration on Light Fastness of Dyed Fibres

Ya. A. Logkun

Zhur. priklad. khim., 33 (July 1960) 1636-1641
The effect of concn. of dye absorbed in lustrous viscose staple fibre on its rate of photochemical degradation has been investigated using two direct dyes (Fast Sky Blue (I) and Pure Sky Blue (II)). These dyes possess protective properties, the light fastness of the dyed fibre increasing with increase in dye concn. on the fibre; in the case of II, its products of fading are stated to act as stabilisers of photochemical processes occurring in the fibre on irradiation. G.J.K.

Variations in the Assessment of Light-fastness Exposures

U. Bülow and S. Horrdin *J.S.D.C.*, 76 (July 1960) 435

Water-repellency Testing—Effect of Shaking Specimens

H. Bundesmann

Melliand Textilber., 41 (Feb 1960) 217-220

Inter-laboratory variations in results of spray tests on the Bundesmann apparatus are shown to be due to differences in the vigour with which different operatives hit the specimen in order to remove surface droplets, even if the same number of blows is administered. Remedial measures advocated are the circulation of test pieces with standard ratings, which would enable each operative to establish the number of blows characteristic for himself required to obtain this rating, or the wider use of mechanical shaking devices, with specific mention of a recent British apparatus. S.M.J.

Behaviour of Sample Pleated Strips of Wool Fabric in an Extensometer. II—Modifications Due to Pleat Curvature and Other Second-order Effects

H. W. Holdaway

Text. Research J., 30 (June 1960) 456-461
The previous model (*Text. Research J.*, 30, 296) was inadequate to account for all observed behaviour of a pleat when extended. Parameters now included take into account radius of curvature at the pleat and variation in fabric stiffness as a function of tension, enabling a very good description of observed behaviour to be obtained. Values of "initial pleat angle" and "initial fabric stiffness" can be determined from experimental results of longitudinal extension and lateral deflection as a function of load. S.B.D.

Synthesis and Properties of Acid Monoazo Dyes (7th Communication) (IV p. 636)

Polarography of Azo Dyes Having a Pyrazoline Component (IV p. 636)

Stiffness of Woven Textiles (VI p. 645)

Determination of Interfacial Forces Between Water-repellent-finished Surfaces and Water and Their Relation to the Water-repellent Effect (X p. 649)

Relation between Thickness and Opacity of Paper (XI p. 654)

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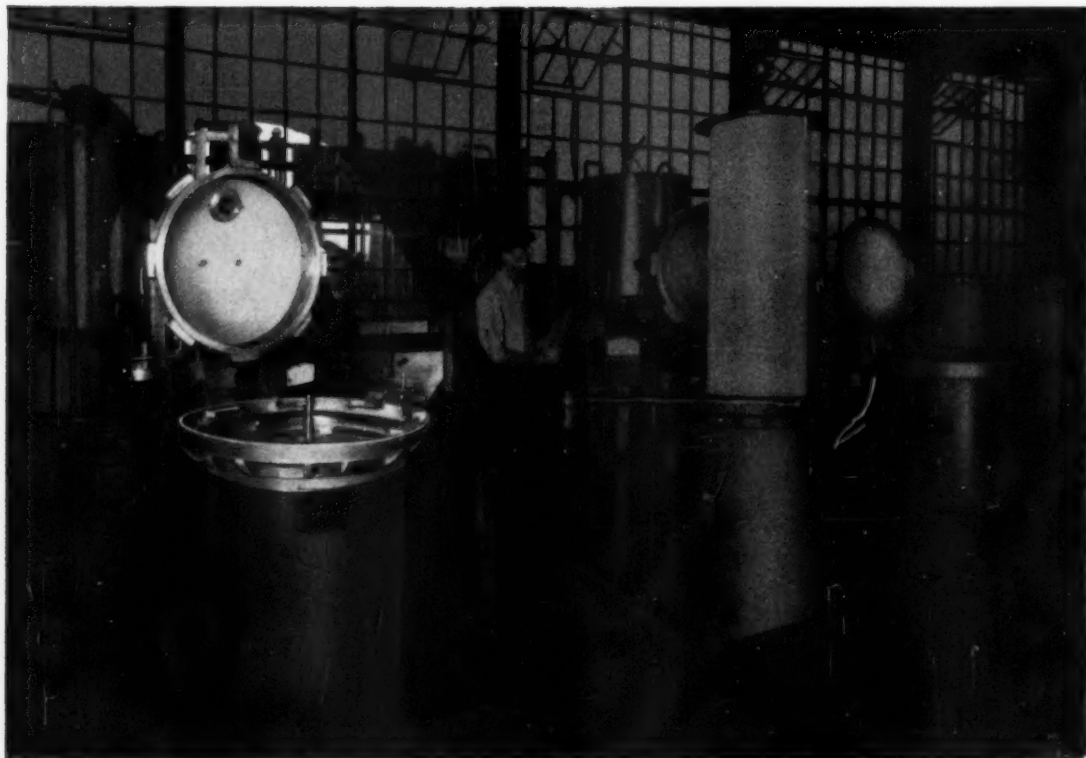
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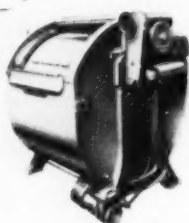
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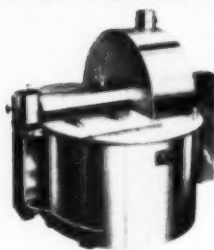
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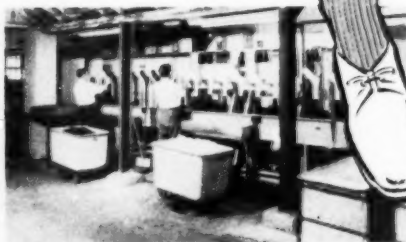


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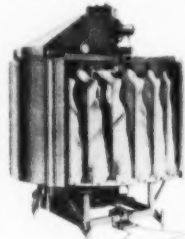
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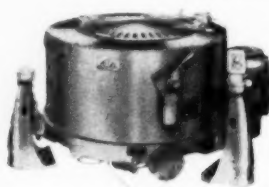


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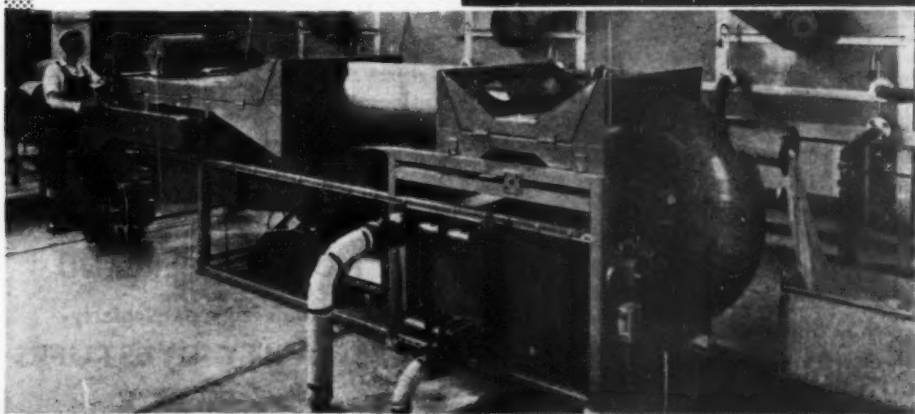
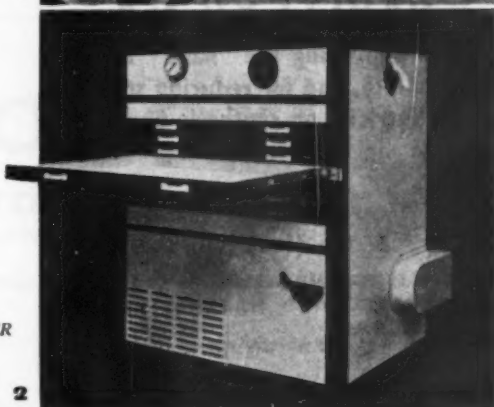
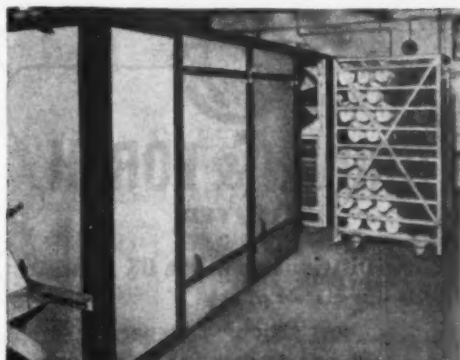
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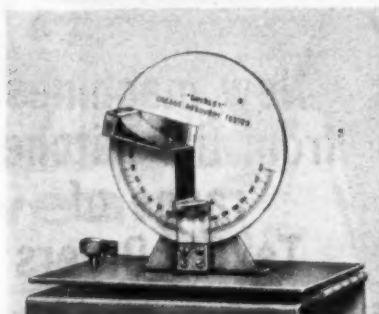
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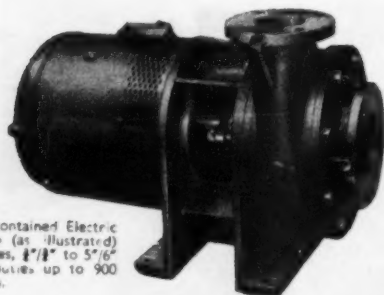
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a new product . . .

is recommended to assist in the dyeing of yarn with fast milling colours, to promote a fine handle to the processed yarn, and avoid unlevel dyeing

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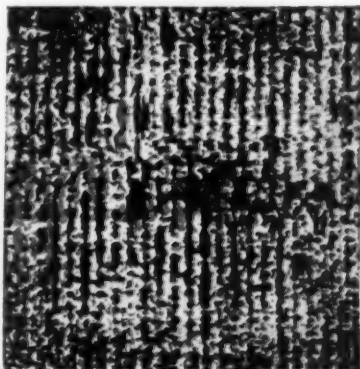
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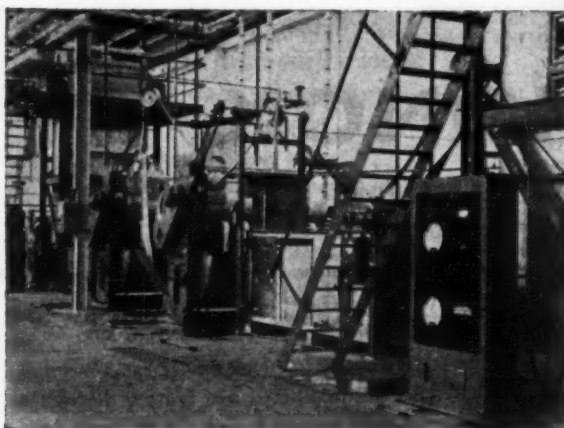
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Continuous Rope Bleaching
Plant. 'J' boxes fitted with
Du Pont type heaters



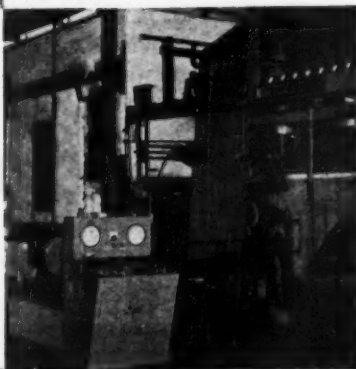
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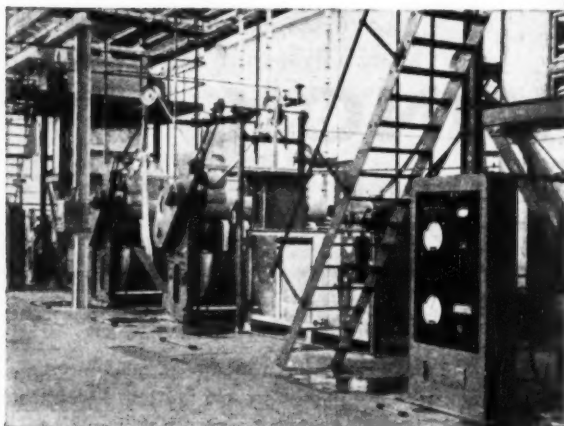
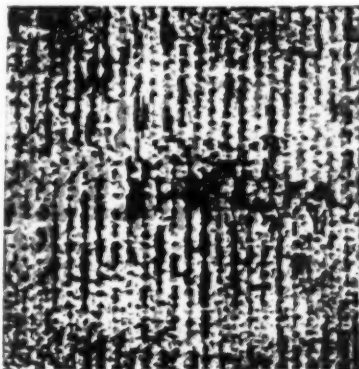
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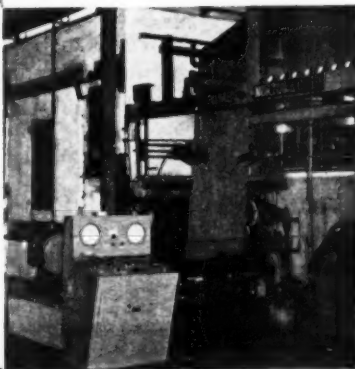
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